



SPECIMENS OF SHELLAC MOULDED ARTICLES

(Indian Lac Research Institute, Nankum)

PRACTICAL APPLICATIONS
OF
RECENT LAC RESEARCH

(REVISED AND ENLARGED)

EDITED BY
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AND
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6 & 7 CLIFFORD STREET, LONDON, W.1
531 LITTLE COLLINS STREET, MELBOURNE, C.1
BOSTON HOUSE, STRAND STREET, CAPE TOWN
LONGMANS, GREEN & CO. INC.
55 FIFTH AVENUE, NEW YORK, 3
LONGMANS, GREEN & CO.
215 VICTORIA STREET, TORONTO, 1

New Edition 1948

PRINTED IN INDIA
AT THE DIOCESAN PRESS, MADRAS
FOR ORIENT LONGMANS LTD.
1948—C2109

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INTRODUCTION TO THE FIRST EDITION

ALTHOUGH lac cultivation in India dates back several centuries, the steadily increasing use of lac in industries began from the year 1900. The exports of lac went up from 2,000 tons in 1900 to 7,500 tons in 1919 and 32,000 tons in 1939. From an output of 2,000 tons in the U.S.A. in 1919, the production of synthetic resins in that country increased to 60,000 tons in 1939. It is apparent that in spite of this phenomenal progress of synthetic resins, the lac industry, instead of dwindling, has also kept on its increased output. Natural and synthetic resins appear to be complementary to each other in most industries, and where the quality difference between the two is small for any particular application, the price factor determines the choice. In view of the increased demand for shellac, it cannot be said that its use has been restricted due to competition from synthetics, and there is no doubt that each is catering to its own fields of use. The fall in the price of shellac is due to the supply exceeding the demand, as in all other commodities, but being an agricultural product, the uncertainty of the quantity produced from season to season and year to year is responsible, in no small measure, for the violent fluctuations in the price. Besides, the cultivation being distributed extensively in small holdings, organised or regulated marketing, which could have stabilised prices, is extremely difficult. While such organisations, necessary to stabilise and improve prices, are contemplated, it is obvious that an extension of its fields of application is more important to achieve the desired end. With this object, the Indian Lac Cess Committee has been fostering researches at Namkum, London and, till lately, in New York. The present brochure is complementary to the publication, *Uses of Lac*, which appeared last year, and more detailed accounts of recent researches are given which may help the establishment of new industries in this country or abroad.

Different sections of this brochure have been compiled by workers engaged in corresponding investigations, and the technical side of this treatise has been carefully scrutinised, and sketches of the more important plants have been included. It is hoped that the present work will fulfil its object.

H. K. SEN

INTRODUCTION TO THE SECOND EDITION

THE value of a raw material increases in proportion to the volume and variety of its uses. Again, the versatility of a material depends on the degree of modifications it can undergo when subjected to different chemical and physical treatments. The natural product lac, the useful commercial form of which is shellac, possesses such a remarkable combination of valuable properties that it finds an important place in the plastics, electrical, adhesive, leather, wood-finishing, hat and several other industries. Recent developments in other natural resins and in synthetic resins threaten to dislodge shellac

more on account of the better performance of the newer products than any economy in their production costs. It is therefore necessary that the range of utility of lac be increased by either finding new uses for the natural product or modifying it to make it more acceptable to the different consumers.

The period 1933-40 saw the development of modified lacs through the combined efforts of the Indian Lac Research Institute, the London Shellac Research Bureau and the New York Shellac Research Bureau, working under a co-ordinated scheme, not to speak of the contributions made by research workers in private firms and laboratories. The main achievements of that period were better plastic moulding compositions through the use of accelerators and hardeners, use of shellac in injection moulding, spirit varnishes with better heat and water-resistance, a method of hot-spraying lac, isolation of hard resin on an industrial scale, etc. The results of those investigations were incorporated in the first edition of this book published towards the end of 1940. The book was well received by both the scientific and technical public and a few industries came into being based on the information given in it. It is to be admitted, however, that some of these newer uses were encouraged by the needs created by the present war and the absence of or difficulty in obtaining certain materials by importation from abroad. At the same time, some of these new industries have been severely handicapped for want of a few modifying chemical agents which used to be imported.

During the last four years the already described recipes for new lac products have been improved, some of them have been altered so that they may be prepared from indigenous materials alone and a few new formulæ have been developed. These have been incorporated in the present edition. The main line of recent developments has been in the direction of flexible and rubbery materials from lac by reaction with fatty acids which have proved useful in many respects.

As regards costing data, normal figures have been given, as a sort of pointer to normal conditions would keep up the original plan and scope of the book. So, the prospective industrialist is warned against undue optimism on seeing the data and is expected to recalculate them on the basis of prices at which he could under the prevailing circumstances obtain the machinery and materials.

Wherever, in the book, a process is indicated as covered by a patent application, the prospective manufacturer is requested to apply to the Secretary, Indian Lac Cess Committee, Ranchi, for the right to adopt the process which would be conceded by the Committee on reasonable and generous terms.

H. K. SEN
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Fig. 1
DHEKIS FOR CRUSHING LAC

CHAPTER I

SEEDLAC

In the manufacture of shellac, seedlac is the starting material obtained by the washing of sticklac. Formerly, seedlac was almost entirely converted into shellac, but as it was found that seedlac when bleached could be used in floor polishes and as a plasticiser in the nitrocellulose lacquer industry, quite a large quantity of this began to be exported to America. The export of seedlac varies now between $2\frac{1}{2}$ and $3\frac{1}{2}$ lakh maunds per year. Of late, the tendency of the consuming trade has been to insist upon seedlac of definitely superior grades so that the cost of bleaching may be the minimum. Ordinarily, as prepared by the time-honoured process, the seedlac is not of high quality due to admixture with sand, wood particles, insect residues, lac dye, etc., unless the sticklac is carefully selected. The country process of final washing with dilute soda solution freshens up the colour of the product to a certain extent, but the main impurities being mechanically mixed, the treatment with soda cannot remove these. The usual process of washing sticklac by rubbing against the sides of a stone vat by feet is efficient to a degree, but the removal of some of the mechanically mixed impurities requires far more intimate rubbing to loosen them than is secured by the present practice. Also, when loosened, the process of washing removes only the lighter impurities, leaving the heavier ones mixed with seedlac. A detailed study of the process was published in the Institute *Bulletin No. 27*, but its introduction required certain modifications to adapt it to existing conditions of lac manufacture, especially in the case of smaller manufacturers. Below is given a description of the process as actually conducted in the Experimental Lac Factory of the Indian Lac Research Institute at Namkum with quantities as large as 5 maunds at a time, but the operation is so simple that it could be equally efficiently conducted with very much larger quantities.

Stage I—Sticklac is first crushed in a stamping mill or by its local equivalent, *dhenki* (see fig. 1) to a size to pass through a 20-mesh sieve. The crushing is best effected by a stamping mill, where power is available, but from actual experience the ordinary country *dhenki* serves the purpose equally well. A unit of five women and two *dhenkis* has been found to be able to prepare 6 maunds of 20-mesh lac in 8 hours.

Stage II—After the sticklac has been crushed to 20-mesh, as described above, it is introduced into stone or cement vats and treated with two maunds of water and $3\frac{1}{2}$ oz. of triethanolamine for each maund of crushed lac. The lac is then washed by rubbing with feet and washing with water as by the usual method.¹ It would be possible to wash about half a maund of lac at a time in vats usually found in lac factories.

When larger quantities are to be washed, or, if facilities permit, a ball-mill (see fig. 3) provided with flint balls can be used for this purpose, worked either by hand or with an electric motor where electric power is available.

¹ Fig. 2 illustrates all the stages in shellac manufacture, in which 8 shows the washing of seedlac in a stone vat.

Stage III—The washed lac is then removed to a cement vat which has a much larger surface area compared with its depth. A four-foot diameter vat with a height of 2 feet would easily handle several maunds in the course of eight working hours. Salt solution, previously prepared by boiling 37 seers of common salt with 100 seers of water and allowing to cool before use, is now poured into the vat. The specific gravity of this solution is 1.2. This salt solution can be used many times by separating the suspended and settled impurities after every operation. The seedlac after being introduced is vigorously stirred in this salt solution by means of a wooden pole for five minutes and the whole allowed to stand. Impurities heavier than seedlac settle at the bottom of the vat, whilst seedlac mixed with lighter impurities floats on the top of the solution. The seedlac is removed by a wooden ladle and placed in contact with clean water in another similar vat and stirred for a few minutes. On allowing the lac to settle, the lighter impurities like wood fibres, animal residues, etc. float on top, whilst purified seedlac settles at the bottom. This operation removes impurities lighter than seedlac. The seedlac is now collected by opening a bottom outlet and is washed free from salt with clean water. This can be recognised by putting a few grains of seedlac on the tip of the tongue. The product is distinctly brighter in colour and purer in quality, and has usually the following analytical figures :

Cold alcohol insolubles	...	6—6.5	per cent
Hot alcohol insolubles	...	1.0—1.8	per cent
Life under heat (at 150° C.)	...	31	minutes
Bleaching test (U.S.S.I.A. method)	...	45—60	c.c.
Colour Index ¹ (Iodine Standard)	...	5—11	

Cost of manufacture of one maund of refined seedlac from scraped lac :

		Rs	A	P
1.8 maunds of scraped lac @ Rs. 10 per maund	...	18	0	0
Crushing of above	...	0	3	9
Washing by feet (one man)	...	0	3	6
Triethanolamine (6 ozs.)	...	0	3	0
Salt treatment	...	0	0	6
Washing by sedimentation	...	0	1	0
Overhead and other charges	...	0	2	0
Total	...	18	13	9

The quality of seedlac is better than that of golden *Baisakhi*.

The price of such a seedlac is expected to be at least 20 per cent higher than that of the commonly exported commodity, and there is no chance of rejection as it may be considered as invariably of such good quality as to pass the American Bleaching and Insolubles Tests.

¹ Fresh *Baisakhi* lac gives a colour index as low as 5, whereas older and mixed samples have given the same as high as 11.

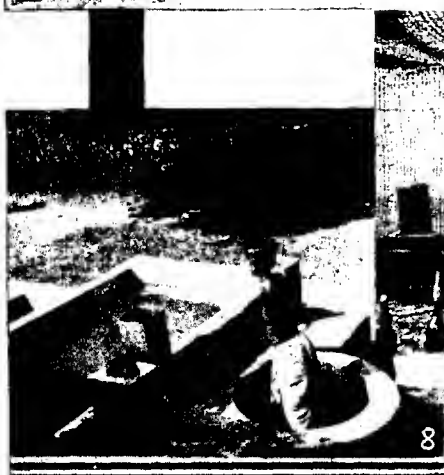
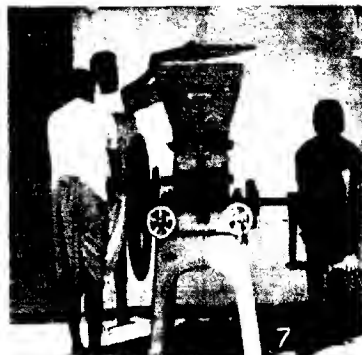


Fig. 2
SHELLAC
MANUFACTURE

CHAPTER II

GARNET LAC

Garnet lac is dark-red lac marketed in the form of pieces of about $\frac{1}{4}$ inch thickness and is distinct from the orange-yellow shellac and button lac. It is prepared by processes different from the country-method used in the ordinary lac factories.

Garnet lac can be made out of seedlac as well as from the by-products of lac factories, like *kiri*, *molamma*, etc. Though the manufacture of high grade garnet lac from seedlac is desirable from the point of view of marketing a purer and standardised product, the possibility of converting the by-products like *kiri* into garnet lac is a more important point to consider.

About 50,000 maunds of *kiri* are produced annually in the course of shellac manufacture and are either exported abroad at a cheap price or stored away for want of a reasonable market. The other by-products are mostly mixed with good shellac for TN manufacture, thereby lowering the quality of the shellac produced. Since these by-products contain a good proportion of lac, they could be worked up for the manufacture of garnet lac with larger profits.

The stages in the process for preparing garnet lac are the dissolution of the soluble lac present in the raw material in alcohol or methylated spirit, the distillation of the clear alcoholic solution in stills to recover the alcohol, tapping the molten lac from the still at the end of the distillation and rolling into thick sheets by passing it between water-cooled rollers.

As regards the working details, they are best described by taking a concrete example, and as the method worked out was with particular reference to *kiri*, the process of working up this material will be discussed at some length.

The equipment required is a crusher (see 7 of fig. 2) for crushing the *kiri*, a number of cylindrical vats (1, 2, 3, of fig. 4) for preparing the solution, a steam-jacketed still (5 of fig. 4) provided with a condenser (16 of fig. 4) for distillation of alcohol, one or two pairs of water-cooled rollers (fig. 5) for rolling the garnet lac into sheets, a second still (21 of fig. 4) for recovering the alcohol from the exhausted material, a rectifying column (fig. 6) for concentrating, from time to time, the weak alcohol obtained at the end of the distillation, and a boiler for supplying steam for heating purposes.

The vats could be made of copper, lined inside with tin, or of well-seasoned teak, and 100 gallons is a convenient capacity. They should be quite alcohol-tight and should be provided with tight-fitting lids and stirrers. The stirrer consists of a wooden rod passing through the lid and is provided with fins at the bottom and a pulley at the top. It could be quite conveniently worked by man-power or a small motor. The vats should be provided also with a narrow pipe ($\frac{1}{2}$ " diameter) fixed at about half the height to

lead the clear solution to the still and a thick pipe (2" in diameter) at the bottom for transferring the exhausted material to the recovery still.

The still is made of copper (tinned inside) and jacketed for steam with mild steel plate. The stillhead is provided with a goose-neck for leading out the alcohol vapours, a stirrer in the centre, a thermometer-hole and an opening for a small steam-pipe. The pipe for letting in the steam is best made of a copper tube about $\frac{1}{2}$ " diameter wound round the stirrer in the form of a spiral and provided with a large number of holes. The still should also be fitted with a gauge-glass about half-way up to indicate the level of the solution.

The condenser attached to the still could be made of coil or pipe pattern, but the latter type is generally more efficient. The rate at which the solution can be conveniently distilled depends on the effective capacity of the condenser and the rate of flow of water through it. The temperature of the alcohol distilled off should not be allowed to rise above 30°C. to avoid any considerable loss of alcohol.

The steam-boiler is the source of heat, and it is not necessary to pass steam at more than 20 lbs. pressure.

Kiri is first broken up into pieces by hand and then coarsely crushed by passing through a crusher (7 of fig. 2). The crushed *kiri* is charged into the vats and treated with one gallon of spirit for every 2½ lbs. of *kiri* taken. A 100-gallon capacity vat will be convenient for treating 2 maunds of *kiri* with the required 66 gallons of spirit. It would be a desirable practice to close the lid of the vat after charging it with *kiri* and adding the alcohol through a small pipe passing through a hole in the lid. This will minimise the loss of alcohol by evaporation. The contents of the vat are then stirred for 1-2 hours and left overnight for the impurities to settle.

The clear solution of lac is then run out to the still to fill it up to not more than $\frac{3}{4}$ of its height. The alcohol is distilled off and collected in suitable drums to be used again for a fresh charge. The temperature (of the vapours above the solution) will be about 78°C. at first and when the greater part of the alcohol is distilled off, it will gradually rise. When about 90 per cent of the alcohol from the solution put in is collected, the rate of distillation considerably slows down and the alcohol from the condenser (16 of fig. 4) begins to come out in a very thin stream, ultimately only in drops. At this stage, the receiver is changed and a small amount of direct steam is let into the still. The distillation is continued for a short while and when again it slows down, a little more steam is let in. The process is repeated until the lac is free from both alcohol and water.

It is important to note that it is not desirable to let water accumulate by letting in too much steam at once. The heating has also to be increased at this stage by increasing the pressure in the steam jacket to 10-15 lbs. The mass of lac becomes very viscous at this stage and requires constant and vigorous stirring.

After steaming a few times, samples of lac are taken out from time to time with a long glass or wooden rod and examined. At first the lac will be soft and dull-looking even on cooling, but when the alcohol and water are driven off, it will be transparent



Fig. 3

BALL MILL

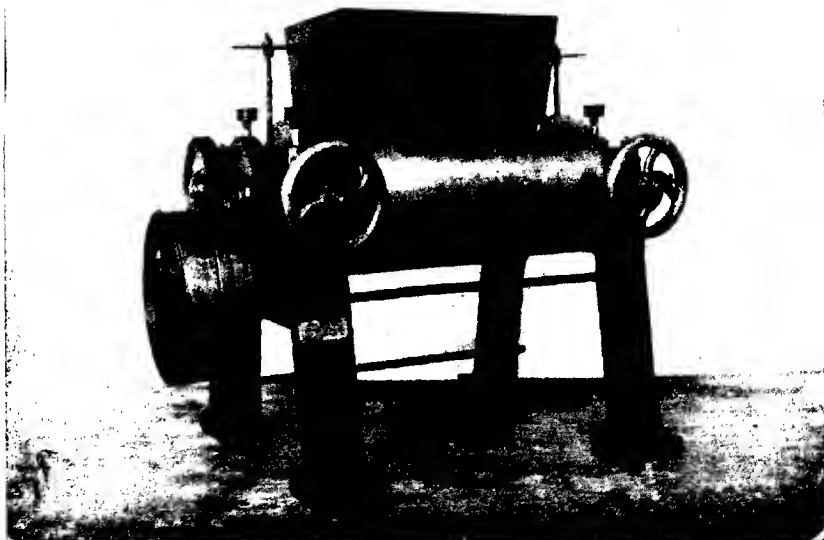


Fig. 5

WATER-COOLED ROLLERS

and crisp. After cooling for a minute or two, the piece of lac should break on bending. Overheating is to be avoided as far as possible.

The lac is now allowed to flow out through the valve (14 of fig. 4) provided at the bottom of the still and can be allowed to collect in blobs. It is, however, preferably rolled into sheets of about $\frac{1}{4}$ " thickness by passing through a pair of water-cooled rollers (see fig. 5) revolving towards each other, or by any other means.

The vat in which the *kiri* was dissolved is now again filled with alcohol, and after stirring for a short while, the impurities are allowed to settle. The clear solution is now let into a second vat and treated with the necessary amount of fresh *kiri* to bring up the strength of the solution to about 12 per cent. The actual quantity could be calculated from the amount of lac in the sample of *kiri* and the amount of lac extracted in the first experiment. The clear solution, after settling the impurities, is run out into the still for distillation. The first vat is treated again with alcohol, and the clean solution is run into the second vat for further concentration. The clear solution now obtained from the second vat is treated with fresh *kiri* in a third vat, and so on. Usually, three extractions are enough to extract most of the lac from *kiri*. The first extraction will yield 40—45 per cent, the second 30—35 per cent and the third 20—25 per cent. 2—3 per cent of lac is usually left over.

When the residue in each vat is extracted thrice or nearly exhausted of the soluble lac, it is run out to a recovery still (21 of fig. 4) and distilled. The weak alcohol in the distillate is concentrated for further use through a rectifier (see fig. 6). The residue in the vats contains, besides the impurities which were originally present in the *kiri*, appreciable quantities of lac wax which could be recovered and used.

The process of alcoholic extraction may be worked profitably either on a small scale with a 25—30 gallon capacity still as in the pilot plant of the Institute or on a larger scale with a still of about 100-gallon capacity or more. The former would give 2—3 maunds of garnet lac and the latter about 10 maunds in a working day of 8 hours. It would be possible to increase the output of the smaller plant by making the process continuous. An idea of the initial outlay and working expenses in the case of the smaller plant can be obtained from the approximate figures given in the statements below.

When materials other than *kiri* and those containing relatively very small proportions of impurities are worked for the manufacture of garnet lac, certain modifications in the procedure, detailed above, would be necessary. In the case of seedlac, for example, nearly twice the amount of material could be taken per gallon of spirit. It would, however, be desirable to add about 2 per cent of a filter-aid like *filtercel* or *kieselguhr* to obtain the seedlac solution by passing it through a filter-press. The filter-press has to be of wooden chambers with the parts of the pump and the press coming into contact with the lac solution made of bronze, stainless steel, etc.; otherwise, the solution will become very dark owing to the action of iron.

In one experiment, working with a good sample of *kiri*, the first extraction gave 42½ per cent, the second about 30 per cent, and the third about 25 per cent; about

2½ per cent being left over in the residues. The percentage of lac recovered in this experiment was 62–63, showing thereby that good samples of *kiri* have a high proportion of soluble lac in them.

The following is the analysis of a typical sample of garnet lac made out of *kiri* in the pilot plant of the Institute:—

Moisture	2.63 per cent
Hot alcohol insolubles	0.22 per cent
Wax	2.22 per cent
Fluidity in seconds (Metropolitan-Vickers method)	660
Life under heat in minutes at 150°C. (modified Westinghouse method)	25
Colour index (Iodine Standard)	19

Approximate Capital Expenditure and Working Expenses for Small Plant

1. INITIAL OUTLAY

			Rs.
1.	Buildings for housing the plant	...	2,000
2.	One boiler	...	2,500
3.	One disintegrator or crusher	...	500
4.	Six 100-gallon teakwood vats with lids and stirrers	...	300
5.	One M. S. 30-gallon steam-jacketed still with surface condenser	...	900
6.	One C. I. 25-gallon recovery still with internal steam coil for heating	...	100
7.	One rectifying column with reflux condenser	...	700
8.	Containers for spirit	...	100
9.	One pump	...	300
10.	One pair of water-cooled rollers	...	600
11.	Erection charges, pipes, lagging material and sundries	...	300
Total Rs.			8,300

NOTE.—Costs of extra building and boiler could be deducted when a small building and a boiler already exist as in the case of most shellac factories of moderate size. A filter-press will not be required if only *kiri* is worked by the sedimentation process described and is, therefore, omitted.

In a few experiments carried out with 2-maund lots of *Kiri* in the Institute semi-pilot plant the following costing figures were obtained reckoning that 2 maunds of *Kiri* would yield 1 maund of garnet lac.

- 1, 2, 3. Wooden vats with stirrers
4. Pipe conveying solution to still
5. Steam-jacketed still
6. Stirrer
7. Main steam inlet
8. Inlets to steam jacket
9. Steam outlet
10. Outlet for condensed steam
11. Coil for injecting steam
12. Level glass
13. Thermometer
14. Pipe for discharging molten lac
15. Connection to condenser
16. Condenser
17. Water inlet to condenser
18. Water outlet from condenser
19. Outlet for condensed spirit
20. Outlet for vat residues
21. Recovery still
22. Opening for leading residues
23. Steam coil for indirect heating
24. Steam pipe for live steam
25. Steam outlet
26. Pipe leading to condenser
27. Discharge man-hole

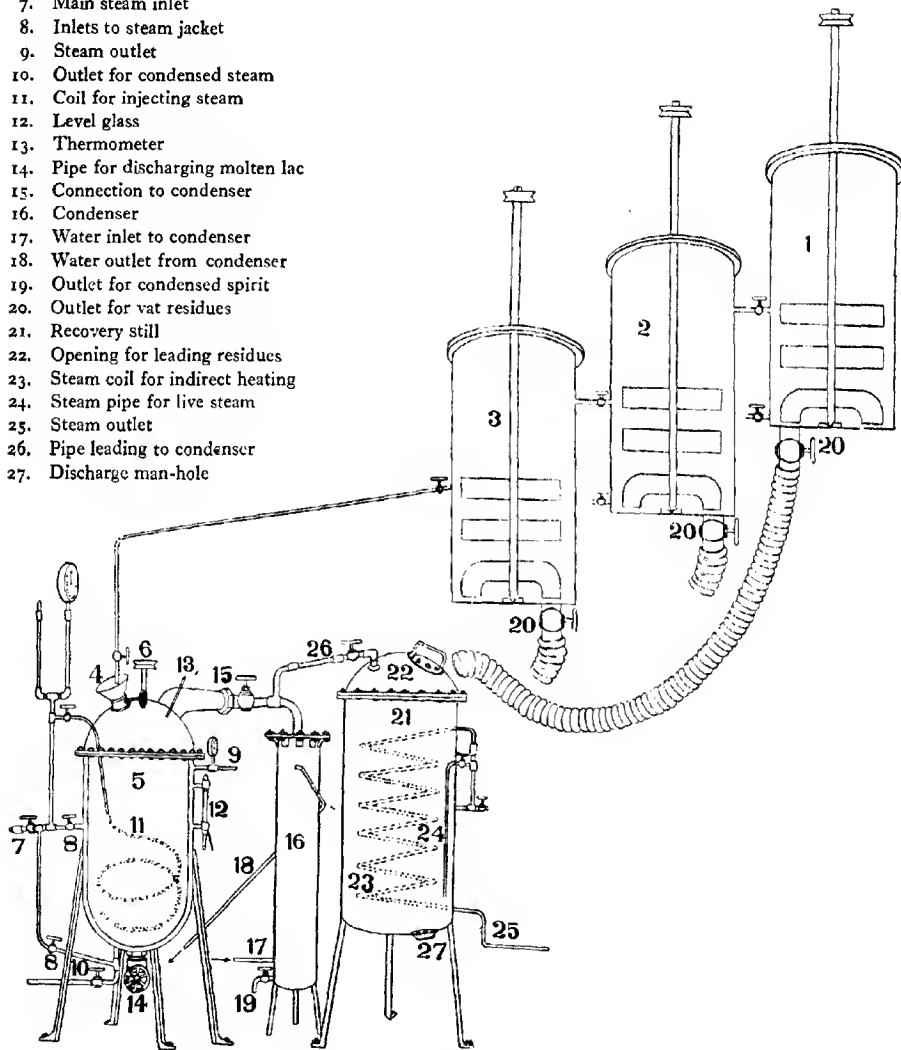


Fig. 4
GARNET LAC PLANT

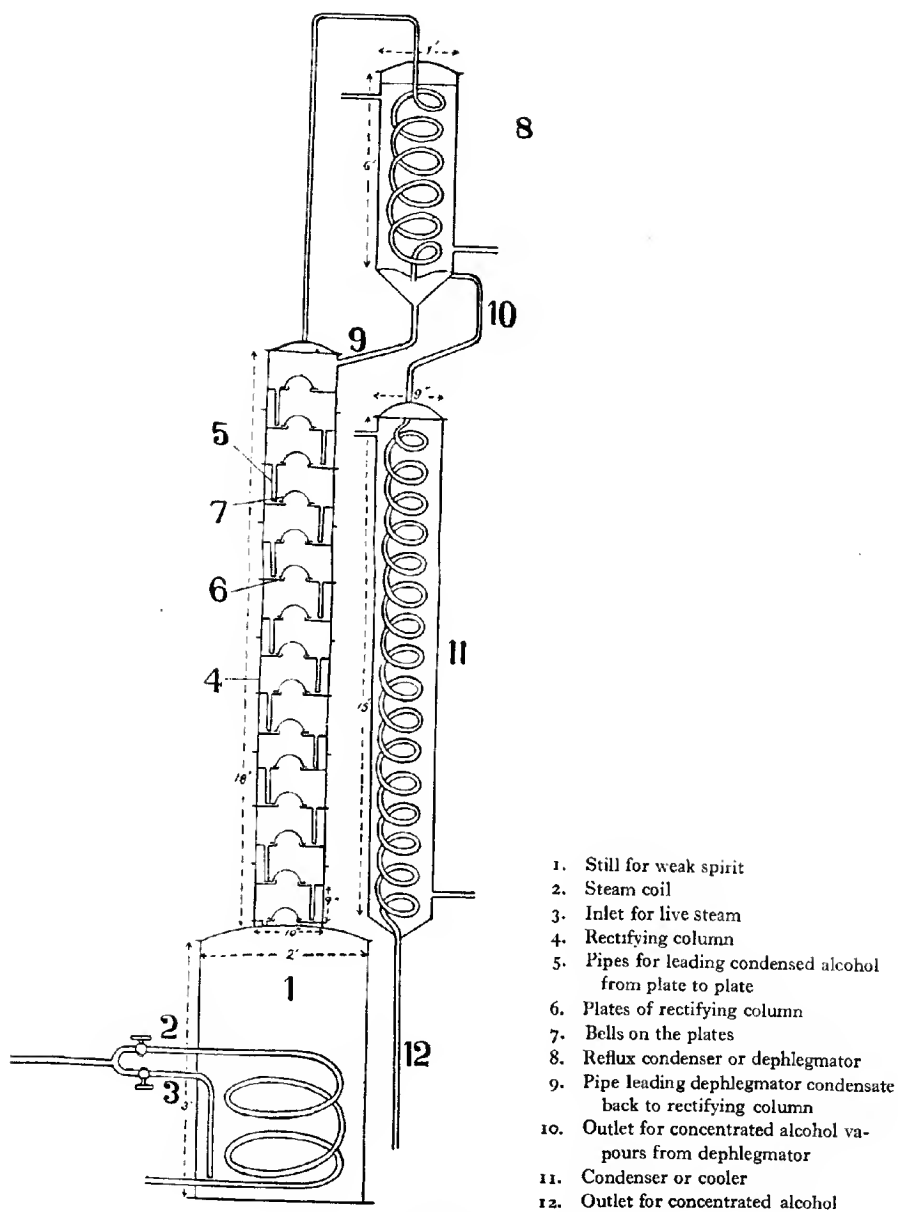


Fig. 6
 STILL AND RECTIFYING COLUMN

2. APPROXIMATE COST OF PRODUCTION OF 1 MAUND GARNET LAC

			Rs.	A.	P.
2 mds. of <i>Kiri</i> @ Rs. 5 per md.	10	0	0
6 gallons of alcohol lost in the process @ Re. 1 per gal.	6	0	0
Labour, fuel, power, etc.	2	0	0
Overhead charges	2	0	0
Total Rs.			20	0	0

From the above figures it may be seen that the loss of alcohol is too great to make the process economical. By a few large-scale experiments and with an efficiently designed plant the loss of alcohol may be reduced to a minimum in which case the cost of production may be brought down to a profitable level.

CHAPTER III

DEWAXED LAC AND VARNISH

Dewaxed shellac is being manufactured by a firm in this country through filtration of an alcoholic solution of lac but the method is not generally known in detail. On account of the increasing interest taken in dewaxed lac, at the present time, a less expensive or simpler method of manufacture would find a wider acceptance.

It has been previously mentioned that garnet lac prepared from Kiri by the process of cold extraction with alcohol is partly dewaxed. The amount of wax left in the lac was found to be of the order of 2 per cent while the particular sample of Kiri from which these garnet lacs were prepared originally contained 4.3 per cent wax. Apart from the fact that dewaxed lac sells at an appreciably higher price than shellac, lac-wax which is obtained as a by-product, is in great demand due to the shortage of carnauba and other waxes for making boot polishes, floor and furniture finishes, etc. The preparation of dewaxed lac from seedlac avoiding prolonged heat treatment has been investigated by two methods—dry and wet.

(a) In the dry method, the seedlac is treated with various solvents for wax. Of these, white spirit is found to be the most suitable. If 20-mesh ground seedlac is treated with four times its weight of white spirit in a ball-mill for 12 hours most of the wax is removed in two operations and the resulting seedlac contains about 0.25 per cent wax. The white spirit can be recovered by vacuum distillation and used over again. Alternatively, seedlac can be extracted by kerosene oil at 50°C. and filtered hot. On cooling the filtrate, wax separates out. By this method only about 2 per cent wax can be removed.

(b) In the wet method, 10 lbs. of sodium carbonate are dissolved in 60 gallons of water in a steam-jacketed enamel pan and in the hot solution are dissolved 100 lbs. of seedlac which is added gradually with stirring. The hot solution (90°C.) is run through a 100-mesh sieve to remove insoluble impurities. The filtrate is allowed to cool to 35–40°C. and 7–12 gallons of sodium hypochlorite solution containing 3% available chlorine¹ are added slowly with stirring. *Baisakhi* seedlac requires about 12 gallons and *Kusmi* 7 gallons of sodium hypochlorite solution. Addition of more bleach does not help in improving the yield of wax. In the absence of bleach liquor the wax does not form a cake and float on the top of the solution. The solution is again heated to boiling and allowed to cool when the wax floats on the surface as a scum. The floating wax is scooped off and the solution is first filtered through a cloth and then passed through a filter-press to remove the wax completely. The wax obtained

¹ The sodium hypochlorite solution used in the process is prepared by passing about 3.5 lbs. of chlorine gas through a solution of 5.3 lbs. of sodium hydroxide in 12 gallons of water. The solution is then tested to contain 3% available chlorine before use.

in the three stages is mixed together and washed in water, melted and cooled in the form of cakes. About 2.1 lbs. of wax are obtained. The filtrate is diluted to four times its volume and precipitated with the minimum quantity of dilute sulphuric acid (about 11 lbs. of strong acid diluted to 22 gallons with water are necessary to neutralise the alkali). The addition of acid is to be carried out slowly and stopped as soon as the solution turns just acid to litmus. The precipitated lac is washed thoroughly till acid-free and dried in a cool well-ventilated room. The dried lac is then melted in a steam-heated pan and stretched on warm tin sheets or heated rollers. After cooling the dewaxed lac is scraped from the tin sheets or rollers in the form of thin flakes. The wax content of the resulting lac is of the order of 0.8—1.0%. 85—90 lbs. of light-coloured shellac is obtained. *Baisakhi* lac gives about 85% yield and *Kusmi* 89%.

In the two processes described above, dewaxed seedlac or shellac is obtained, but if a dewaxed solution of lac is required, the process, which does not require any plant other than that ordinarily available in a varnish factory, is as follows.¹

(c) Wax containing shellac varnish is mixed with white spirit (sp. gr. 0.7924), at 20°C. and the mixture is gradually heated to 70–80°C. over a period of half an hour (the use of steam-jacketed vessel is preferable, but hot water or steam coils may also be used for heating). The heat is then turned off and the mixture is allowed to cool and settle. The wax particles swell and rise to the surface of the varnish with the white spirit, and the clear varnish can be run off from the bottom of the vessel after 24 hours. The yield of clear varnish is between 85 and 90 per cent.

The white spirit containing the shellac wax can be re-used at least three times for dewaxing fresh batches of varnish, and the shellac wax can be recovered by distilling off the white spirit, or the mixture can be sold as an ingredient for white spirit wax polishes. Ordinary liquid paraffin may be employed in place of white spirit, and gives equally satisfactory results.

Where it is neither possible nor desirable to heat the varnish, the whole process can be carried out in the cold, but the time required is then considerably longer. After mixing the white spirit with the lac spirit varnish, at least 48 hours will be required for the wax to separate.

It has been found that for rapid separation, the concentration of lac in the varnish is critical, 3 lbs. of lac to a gallon of industrial methylated spirit (61 O. P.) being the optimum, and 3 volumes of varnish are treated with 1 volume of white spirit; lower or higher concentrations than a 3-lb. 'cut' to the gallon require longer time for the wax to separate. For example, 28, 36, 40 and 48 hours are required for the wax to rise to the surface when varnishes of 2½, 4, 5 and 6 lb. 'cut' per gallon respectively are treated as described, with white spirit, employing the warm method. For purposes of ready reference the specific gravities and solid contents per cent of such solutions are

¹ This process has been worked out at the London Shellac Research Bureau.

given below.

Cut per gal. in lbs.	S. G. at 15.5°C.	Solid content per cent.
3	0.9030	31
4	0.9242	37
5	0.9427	42.5
6	0.9588	47

MANUFACTURE OF DEWAXED LAC AND LAC WAX BY PROCESS (b)

A. *Capital Expenditure*

	Rs.
Building	5,000
2 Wooden vats fitted with steam coils and stirrer: 500 gal. capacity each	1,000
4 Acidifying Tanks with stirrer; 800 gals. each	1,600
1 Cement Tank lined with Porcelain for bleaching solution; 150 gals.	800
2 Washing Tanks of 800 gals. each	800
2 Filter Presses	2,500
Other accessories	300
	<hr/> Rs. 12,000 <hr/>

B. *Cost of Manufacturing 800 lbs. of Dewaxed lac and 20 lbs. of
Lac wax.*

	Rs.	A.	P.
Seedlac, 940 lbs. at 4 as. per lb.	235	0	0
Sodium carbonate, 120 lbs. at 1 anna per lb.	7	8	0
Caustic Soda, 53 lbs. at Rs 10 per cwt.	4	11	9
Chlorine gas, 35 lbs. at 6 as. per lb.	13	2	0
Sulphuric acid, 110 lbs. at Rs. 5 per cwt.	5	0	0
Labour and overhead charges	10	0	0
Depreciation, Interest, etc. 15%	6	0	0
	<hr/> 281	5	9
Less cost of 20 lbs. lac wax at Rs. 2-8-0 per lb.	50	0	0
	<hr/> Rs. 231	5	9
Net cost per maund or 80 lbs. of Dewaxed lac:	<hr/> Rs. 23	2	0

CHAPTER IV

BLEACHED LAC

The manufacture of bleached lac is an important industry, especially in the U.S.A., where about 300,000 maunds of it are produced for the pale varnish and nitrocellulose lacquer industries. The operation is beset with many technical details which have been worked out with great care by several factories in the U.S.A. to obtain the finest product. These details are trade secrets, although the general process of bleaching is well-known. In the following pages, details of the process as worked out at the Indian Lac Research Institute have been recorded, and in view of the facts that 30—35 per cent of the total export of lac is converted into bleached lac and attempts to introduce other simpler or more efficient processes are continuing, a short historical introduction to the subject has been included for the information of the interested manufacturer.

Ordinarily, for the manufacture of bleached lac, high-grade seedlac is almost exclusively used, although in the past the starting material was shellac. The change-over from shellac to seedlac has taken place on account of the higher cost of shellac as compared with the lac material in seedlac, although, as will be seen later, the amount of bleach liquor consumed with shellac is considerably less than with seedlac. There is another factor which accounts for the suitability of seedlac for the purpose—the absence of orpiment or rosin (usual adulterants in shellac) which, while consuming larger quantities of bleach liquor, yields inferior products.

There are three processes for bleaching—(i) *Physical*, in which the colouring matter is removed from the alcoholic solution of lac through adsorption by animal charcoal or by the action of sunlight; (ii) *Electrical*, in which the colouring matter is bleached by nascent oxygen or chlorine, liberated at the anode; and (iii) *Chemical*, in which the colouring matter is destroyed by oxidising agents such as chlorine and hypochlorous acid.

The Physical Method consists in dissolving lac in alcohol and boiling the solution with well-burnt and freshly heated animal charcoal or with activated carbon until the filtrate is nearly colourless; the alcohol is then distilled off, leaving the bleached product behind. But this process causes only a partial decolourisation of lac. The decolourisation can also be effected by treating the lac with an aqueous emulsion of cocoanut oil or other fats, accompanied by agitation and heating. The lac is then separated after cooling, and dissolved in alcohol. The dirt and the excess of fat are separated by filtration. These methods are very costly and the lac obtained in these ways is somewhat altered in its physical properties. Bleaching by sunlight is an ancient practice, but is impracticable, both on account of the inefficiency of bleaching and the long period necessary to complete the process.

The Electrical Method which involves the electrolysis of an alkaline solution of lac is the subject of a German patent, the salt used being sodium chloride with or without the addition of sodium fluoride, the active bleaching agent being, of course, the liberated chlorine. The machinery is intricate and the process is of doubtful practical utility. In another process, the lac is peptised in an aqueous solution of sodium carbonate (2—3 per cent solution) at about 80°C.; to this is added a small quantity (1 or 2 grams) of caustic potash; an electric current is passed through the solution for 8 to 16 hours at a current density of 0.01 amp. per sq. cm. at a solution temperature below 70°C. and the bleaching is effected by means of the 'nascent oxygen' separated at the anode. If a suitable amount of sodium chloride (common salt) is mixed in the peptised solution, the electrolysis is facilitated. Here as only a mild-bleaching agent is used, the finished article is pure in quality and does not deteriorate when stored for a long time. Another advantage is that the cleaning operation during manufacture is simplified.

The Chemical Process involves the use of oxidising agents such as chlorine, hypochlorous acid, oxygen, etc. The earliest method was to bring lac into contact with an acidified solution of bleaching powder for some time; it was then washed, kneaded in hot water, placed back into the chloride of lime solution and bleached. It has the drawback that through the treatment with chloride of lime solution the bleached lac sometimes loses its solubility in alcohol, which, however, can be restored if it is moistened with a little ether (1 part to 20 parts of shellac) in a closed vessel for several hours till it swells. The lac thus treated becomes perfectly soluble again.

P. Stuhlman in a U. S. Patent describes the following method:

The lac, more or less finely divided, is dissolved in a cold, dilute alkaline lye which is kept in continuous motion by stirring. The usual bleaching agent, such as hydrogen peroxide, sodium perborate or others, can be added to the lac during solution. Formaldehyde or furfural together with the bleaching agents effects bleaching of the lac solution and also hardens it at the same time when carried out in the cold. The action can be assisted by adding phenol or other hardening agents as well. Very good results are obtained when the lac is dissolved with the addition of sodium bicarbonate and is bleached and hardened with formaldehyde and hydrogen peroxide.

E. Dutt in a British Patent describes the bleaching process with Indian bauxite: Lac is dissolved in methyl or ethyl alcohol (10—15 per cent solution) and filtered. The filtrate is neutralised with ammonia, amines or oxides of the alkaline earth, such as calcium oxide, barium oxide, etc. The neutral solution is then treated with calcined aluminous earth (Indian bauxite) containing as little silica and iron oxide as possible and containing a high percentage of titanium oxide. If one of the oxides of alkaline earths were used to neutralise the solution, carbonic acid gas is passed through the solution to precipitate the alkaline earth compounds as carbonates and the solution is refiltered. It is then treated with water to precipitate the shellac.

The modern process, extensively practised in Europe and America, aims at attaining a great uniformity in the finished product and consists of operations which are numerous, distinct and equally important and are conducted with great precision. The factors which influence the process of bleaching are discussed below :

Lac should be fresh and of the purest kind available and free from lac-dye. Mechanical impurities, such as insect bodies, fragments of wood, sand, etc., must be effectively removed. The more completely the interfering soluble matter is washed and removed, the more is the improvement in the bleaching quality of the lac. The nitrogenous matter present in lac is mainly responsible for poor bleachability and increased consumption of bleach liquor. Old lac, which has not been thoroughly washed, is bleached with difficulty and sometimes the desired lightness of colour is not attained whatever be the amount of bleach liquor added. Lac containing a small amount of dye that has become 'set' with age demands a greater quantity of bleach liquor.

It has been observed that the dye mostly consists of pigmented protein containing iron. It is essential to remove this before seedlac is dissolved in sodium carbonate solution for bleaching. To accomplish this, seedlac is first boiled in 1 per cent alcoholic water once or twice depending upon the quality of lac. Boiled water extract containing the impurities is thrown away. By this treatment, the major portion of water soluble non-resinous matters are removed and the resulting lac dissolves easily in sodium carbonate solution. This treatment is advantageous for two reasons, (a) total time required and total available chlorine consumed for complete bleaching are appreciably reduced and (b) due to softening action of hot water, lac resin is reconditioned with the result that it shows improvement as regards its keeping quality after bleaching.

The sample of seedlac or shellac should be coarsely ground so as to pass a 10-mesh sieve, as it has been observed that fine grinding means more bleach liquor, possibly due to more nitrogenous matter being brought into solution from a finely ground material. The lac should be dissolved with as little soda as possible for making complete solution with ten times the weight of water; higher concentrations of soda than necessary will mean a greater quantity of bleach liquor and give bleached lac of better keeping quality but of poorer colour. The lac should be dissolved in soda solution at about 80°C. with constant stirring as quickly as possible. In any case, it should not extend beyond one-and-a-half hours, as the longer the period employed in dissolving the lac, the greater will be the quantity of bleach liquor consumed. By dissolving lac in alkali at higher temperatures than 80°C., a larger quantity of bleach liquor becomes necessary. The hot soda solution of lac should be strained through a fine muslin cloth with great care, as the insolubles are materially responsible for the higher bleach liquor requirement. These insolubles mostly consist of proteins and other nitrogenous substances which consume substantial quantities of bleach liquor. Some samples of Burma and Siam lacs remain practically unbleached and show a green colour, however much of bleach liquor may be added. But if, before bleaching, the lac solution is filtered through

filter paper, then a well-bleached solution can be obtained. Hence filtration forms an essential condition for good bleaching.

It has been established that the bleach liquor acts best when the temperature of the soda solution of lac is 35°C. A higher temperature of course shortens the time of bleaching, but it will affect the keeping quality of the resultant bleached lac. The rapid addition of bleach liquor or its addition in one lot gives a bleached lac with poor life and sometimes of poorer colour. The first addition of bleach liquor may be made with approximately half the calculated quantity required, but successive additions are made in small amounts. Each addition of bleach liquor should be allowed to exhaust itself as completely as possible before the next addition, as judged by testing with starch-iodide paper which should turn only faintly blue when a drop of the liquid, being bleached, is placed on it. (The starch-iodide paper is a filter paper impregnated with starch and potassium iodide solutions and then dried in a dark place, preferably in vacuum.) Care should be taken not to treat the lac with too much bleach liquor, as it might be damaged by overbleaching and the resultant bleached lac will soon become insoluble in alcohol.

For a rapid and approximate determination of bleach liquor required by a sample of lac, the following method is used: Add 10 ccs. of a 5 per cent soda solution to 2 grams of lac in a test tube. Immerse the test tube in a tall beaker containing water maintained at the boiling point. Keep on stirring the contents of the tube for an hour by means of a glass rod. At the end of this period, strain the solution into a test tube through a muslin cloth. Press out the last drop of the solution adhering to the insolubles. Keep the test tube in a beaker containing water at 40°C. Add from a burette 2 ccs. of bleach liquor containing 3 per cent chlorine. After the bleach has been consumed as tested by a strip of starch-iodide paper, add more bleach, 1 cc. at a time, until the colour of the solution of the sample matches with the colour of the iodine colour standard (0.0005 N iodine solution)¹, the comparison of colour being made in test tubes of same diameter.

When the bleaching is finished, the lac is precipitated with a 2—5 per cent solution of sulphuric acid, but when precipitated with acetic acid, the bleached lac appears to have longer life. Precipitation on the slightly alkaline side always gives a product with long life, but if definitely alkaline, a certain amount of loss in the yield is incurred. Precipitating on the acid side yields a product with comparatively poor life.

The gradual formation of insoluble lac is due to the residual mineral acid and chlorine which have not been removed during washing. To get rid of these difficulties, the lac should be precipitated in a fine granular form by slowly adding 2.5 per cent

¹The solution of iodine used as a standard has a strength of 0.0005 N and is very sensitive to light. So a decinormal solution of iodine is first made as follows: 12.7 grams of iodine are added to a strong solution of 25 grams of potassium iodide which, when dissolved, is made up to 1000 ccs. This solution is stored in a dark place. 1 cc. of this solution made up to 200 ccs. gives the iodine colour standard.

sulphuric acid with continuous stirring by means of a mechanical stirrer and washed with several changes of cold water until the washings are neutral to litmus. As common salt and sulphate of soda are the by-products of the process, these should be fully washed out and the only way to ensure this is to test the final wash water for sulphates and chlorides by barium chloride and silver nitrate respectively. Often these inorganic matters are the cause of insolubility of the bleached lacs which are not properly washed. The excess of unreacted chlorine can be removed by adding a little hydrogen peroxide before precipitation. Thorough removal of mineral acid and absorbed chlorine can be effected by wet-grinding the material in presence of 1 per cent alcoholic water in an edge runner mill or a ball mill. The suspension is subsequently hydro-extracted and washed in a centrifuge with plain water.

The precipitated bleached lac is squeezed out as free of water as possible and spread on a glass plate or a sheet of white paper. It is then vacuum-dried or dried by passing a warm draught of air at 30°—35°C. or simply in a dark well-ventilated room until the moisture content is reduced to about 2 per cent, as, otherwise, bleached lac will be blocked on storage. Bleached lac is usually marketed with a maximum moisture content of 6 per cent.

Any heat treatment during the process of manufacture is detrimental to the life of bleached lac. Storing bleached lac at room temperature, especially in tropical countries, has a deleterious effect on its life. Strong light also has an appreciable effect on the keeping quality of the bleached lac. It may be stored in a cool, dry and dark place at 16°—18°C. in glass, earthenware or wooden vessels, preferably under water. Bleached lac varnishes deteriorate in colour even when stored in glass containers, which, however, may be considerably checked by storing at 5° to 10°C.

In preparing bleach liquor, care should be taken that there is a trace of calcium salt left in the solution which should be as neutral as possible. An excess of alkalinity makes it more difficult to bleach poor quality lacs in spite of an excess of bleach liquor, and yields a bleached lac of high acid value. The bleach liquor should have a total alkalinity between 0.5 and 0.7 N. The bleach liquor containing 3 per cent available chlorine is best suited, as higher chlorine content will deteriorate the final product. Pure bleaching powder containing over 40 per cent or preferably 65 per cent chlorine should be used for the preparation of bleach liquor. Ordinary laundry bleaching powder is of no use.

Bleach liquor may be prepared by the following methods :—

- (a) Triturate about 50 grams of bleaching powder containing 65 per cent available chlorine with small portions of water and transfer them to a big beaker, until a volume of 600 ccs. is obtained. Now add a 15 per cent solution of sodium carbonate (containing 58 per cent soda ash) portionwise and with stirring, to the bleaching powder solution, until a portion of the filtered bleach liquor gives but a faint precipitate of calcium carbonate on addition of a few drops of sodium carbonate solution. Filter out the calcium carbonate and make up the filtrate

to one litre with water. Determine the available chlorine content of this solution by standard sodium thiosulphate. Then carefully dilute to 3 ± 0.05 per cent available chlorine content.

- (b) Prepare a normal solution of caustic soda by dissolving 40 grams of caustic soda in one litre of distilled water. Pass dry chlorine gas into this solution until a drop of phenolphthalein added to a portion gives a pink colour which disappears immediately. The chlorine content is determined by standard sodium thiosulphate and the solution is diluted to 3 ± 0.05 per cent available chlorine. Since the available chlorine in bleaching powder goes on decreasing when stored, especially under tropical conditions, this method would be more suitable.

All the chemical reagents used in the bleaching operation act on vessels made of iron, aluminium and other metals and cause generally darkening of colour of the resultant bleached lac. Even a minute trace of dissolved iron present in the bleached lac causes cloudiness in the final alcoholic varnishes and lacquers. Hence glass or enamelled iron vessels are recommended, and for large-scale production cement vats lined with glazed tiles would be satisfactory.

Manufacture of Bleached Lac

750 grams of bleaching powder containing 40 per cent available chlorine are dissolved in 3 litres of water contained in an enamelled iron vessel or glass vessel by stirring. 600 grams of sodium carbonate (containing 58 per cent soda ash) are dissolved in 2 litres of water in another similar vessel. About $4/5$ ths of the sodium carbonate solution is added to the bleaching powder solution with stirring. The sludge is allowed to settle down and the supernatant liquid is decanted off into another glass or enamelled vessel. A few drops of sodium carbonate are added and if the decanted solution still gives a precipitate, the addition of sodium carbonate is continued until only the faintest turbidity is obtained. The sludge is allowed to settle and the clear liquid above is decanted. The sludge is stirred up with another 2 litres of water, allowed to settle, and the clear liquid above again decanted. The operation is repeated twice. The decanted liquids are mixed together and made up to 10 litres with water. If the operation has been carried out carefully, this solution will contain 3 per cent available chlorine.

2000 grams of lac powdered to pass 10-mesh sieve are added gradually with stirring to a hot solution of 250 grams of dry sodium carbonate in 20 litres of water which is maintained at 70° — 80° C. After an hour and half, when all the lac has been dissolved, the solution is strained through muslin cloth in order to remove impurities. This lac solution is then maintained at 35° C. and bleach liquor in lots of 500 ccs. is added till the solution is nearly but not completely bleached. Thereafter the addition of bleach liquor should be made in smaller quantities until the end-point, as described below, is reached. A few ccs. of the lac solution undergoing bleaching is diluted with

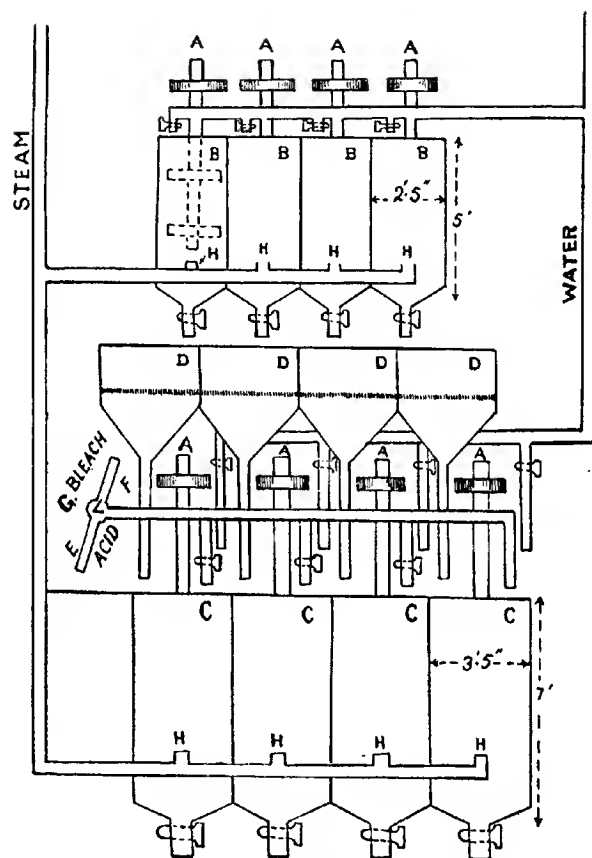


Fig. 7
PLANT FOR BLEACHING LAC

water until the volume in ccs. is about 15 times the weight of lac in grams. The colour of this solution should equal as closely as possible to that of the standard iodine solution of 0.0005 N strength when the comparison is made with test tubes of equal diameters. The successive additions are made when a starch-iodide test paper turns only faintly blue when a drop of the liquid being bleached is placed on it. The bleached solution should not be allowed to remain longer in contact with free alkali than is absolutely necessary for bleach consumption, as longer contact with alkali darkens the lac. The bleached lac solution is then diluted with an equal volume of cold water and dilute sulphuric acid (concentrated acid diluted with about 40 times its volume of water) is added in a thin stream with rapid mechanical stirring, in order to obtain bleached lac in a fine granular form which is easy to wash. The addition of acid is stopped when the solution is just neutral to litmus. Excess of acid should be avoided as the life of the bleached lac obtained will then be short and it will become insoluble in alcohol in a short time. The precipitated bleached lac is suspended in a muslin bag and thoroughly washed in a stream of cold water. The washing should be stopped when the filtrate shows no change of colour on litmus. Water is then pressed out of it and the bleached lac is spread in thin layers over glass plates or sheets of white paper and allowed to dry in a well-ventilated room or preferably under the fan away from direct sunlight. The drying may be effected in about 24 hours in a dry atmosphere under a fan. It should be stored in a glass or enamelled vessel in a cool dark place, preferably in a refrigerator at a temperature of 5°-10°C.

Large-Scale Bleaching of Lac

The materials and the process of operation are the same as described above. The lac is dissolved in a battery of cylindrical cement tanks, B (fig. 7), lined with glazed tiles, each tank being 5 ft. high and 2.5 ft. in diameter. One maund of lac is handled in each tank. First, the requisite quantity of water is let into the tank and the calculated quantity of soda is added. The requisite temperature is maintained by letting in steam through steam inlets, H. The tanks are provided with wooden stirrers, A, worked by power. Seedlac or shellac in a free granular condition is added into each tank. After the dissolution is complete, the solution is run out from the bottom into a set of strainers, D. The strained solutions are transferred to a battery of bleaching tanks, C (with glazed tile lining), of diameter 3.5 feet and height 7 feet. Steam is again used here to maintain the requisite bleaching temperature (35°C.). The bleaching solution is let in through a stoneware pipe, F, at a predetermined rate, into the tanks. The acid used for the precipitation of bleached lac can be let into the tanks through the pipe, E, by manipulating a three-way valve, G, after a preliminary flushing with water. The precipitated bleached lac is drawn out on trays with close-woven muslin bottoms and the washing with cold water is done on the counter-current principle. If the precipitated bleached lac happens to be in large lumps, these should be ground in a porcelain lined ball-mill to fine granules after a preliminary wash, before continuing further washing.

Bleaching of Kiri

Kiri, the chief by-product of lac manufacture, can be bleached after freeing it of nitrogenous materials. To do this, the *kiri* is extracted with alcohol and filtered so that most of the protein matter, being insoluble, is removed from the solution. The alcohol extract is evaporated to a syrup to remove most of the alcohol and poured into a large volume of water. The precipitated resin is filtered off, dissolved in sodium carbonate and bleached in the ordinary way.

Classification of Lac according to Bleachability

Kusum seedlac or shellac is best suited for bleaching operation and produces the maximum lightening of colour with a small consumption of bleach liquor. *Palas*, *ber* and other seedlacs, with the exception of their best quality, require more bleach liquor and yet do not bleach to the same lightness of colour. Burma and Siam seedlacs and TN shellac containing too much orpiment are unsuitable for bleaching. Seedlacs, in general, consume more bleach liquor than their corresponding shellacs.

100 gms. of average *Kusmi* seedlac generally consume 160 ccs. bleach.

"	"	"	"	shellac	"	"	130	"	"
"	"	"	"	<i>Baisakhi</i> seedlac	"	"	300	"	"
"	"	"	"	shellac	"	"	260	"	"
"	"	"	"	fresh <i>Baisakhi</i> seedlac prepared by triethanolamine washing and salt floatation method of the Institute			require only 165	"	"

The consumption of bleach liquor varies within wide limits for seedlacs of same quality but of different localities. Analytical results show that *Kusmi* seedlacs take bleach liquor varying from 30 ccs. upto 53 ccs. and *Baisakhi* seedlacs similarly consume 51 ccs. to 113 ccs. for 30 grams of seedlac.

Seedlac may be divided into three bleaching grades :

- (1) Those that bleach readily to a standard lightness of colour on addition of less than 70 ccs. of 3 per cent bleach liquor for 30 grams of seedlac, *viz.*, *Kusmi* and best type of *Baisakhi* seedlacs.
- (2) Those that consume more bleach liquor and reach nearly the same standard lightness of colour, *viz.*, *Baisakhi*, Assam and other Indian seedlacs and the best type of Burma seedlac.
- (3) Those that do not attain standard lightness of colour even when considerable quantities of bleach liquor are added, *viz.*, Siam and Burma seedlacs and inferior shellacs.

If the lac is bleached in presence of quinoline or pyridine, the resulting bleached lac has better life under heat and keeping quality. It is also found that bleached lac varnish in which dioxane is used as the solvent does not suffer esterification and consequently its drying property is unimpaired unlike varnish prepared in alcohol.

The storage or keeping quality of bleached lac depends upon the following factors. (1) the quality of lac and the treatment it has undergone previous to bleaching, (2) the methods used for dissolving the raw lac, (3) the manner of carrying out the bleaching operation, (4) the extent of the neutralisation and the nature of the acid used in precipitating the lac, (5) the influence of antichlors, (6) the methods of washing and stirring the finished product, and (7) the manner of storage.

The consumption of bleach liquor depends upon the following factors : (1) the quality of lac, *viz.*, *Kusmi*, *Baisakhi*, etc., (2) the purity of lac, *i.e.*, lac free from dirt, lac-dye, nitrogenous matters and other impurities, (3) the age of lac, (4) fineness of grinding, (5) the quantity of sodium carbonate used in dissolving the lac, (6) the temperature at which solution of lac is made, (7) the time allowed for making the solution of lac, (8) the filtration of the solution of lac, and (9) the alkalinity of the bleach liquor.

Manufacture of Bleached Lac

	Rs.
A. Capital expenditure on vessels for producing one maund of bleached lac ...	1,500
B. Cost of preparing 1 md. of bleached lac—	
1.1 mds. of seedlac @ Rs. 20 per md. ...	22 0 0
33 lbs. bleaching powder @ Rs. 7 per cwt. ...	2 1 0
46 lbs. sodium carbonate @ Rs. 7 per cwt. ...	2 14 0
11 lbs. sulphuric acid @ Rs. 5 per cwt. ...	0 8 0
Labour and overhead charges ...	3 0 0
Total cost ...	<u>Rs. 30 7 0</u>

CHAPTER V

HARD LAC RESIN

When shellac is exhaustively extracted with ether, the ethereal solution contains a soft material called the 'soft resin,' whilst the insoluble portion, which has a higher softening point than shellac, is called ' α -lac' (according to the Institute nomenclature), or pure resin. This separation is, however, incomplete by other processes, when varying quantities of soft lac are associated with pure resin, giving rise to a product which has also a higher melting point than shellac and is technically known as hard lac resin. In recent years, the manufacture of hard lac resin from shellac and seedlac has been assuming considerable importance in the varnish and electrical industries, chiefly on account of the fact that it has got many improved properties, especially with regard to heat and water resistance, better adhesion and elasticity and 'non-greening' on copper. Several methods have been developed for preparing the hard lac resin. These are (1) separation of hard lac resin by polymerisation with urea in the cold in acetone solution, (2) by fractional precipitation of hard and soft lac resins from acetone solutions at low temperatures, (3) by direct liquid extraction with toluene, benzene, trichlorethylene, etc., (4) by fractionating lac into soluble and insoluble components with hot alkaline solutions of sodium carbonate, borax, etc. and (5) by cold extraction with ethyl acetate. Of the above, methods (1), (2) and (5) have been investigated at this Institute, and the other two at the London Shellac Research Bureau.

Below is given a brief description of the different methods :

(1) SEPARATION OF HARD LAC RESIN BY POLYMERISATION WITH UREA IN THE COLD IN ACETONE SOLUTION

A 40 per cent solution of shellac or seedlac in acetone is made and 7-8 per cent of finely powdered urea (on the weight of lac) is then added to the shellac solution with stirring until all the urea is dissolved. The vessel containing the solution is then closed air-tight and kept at room temperature (25°-30°C.) for 4 days. During this period, the solution sets into a hard mass which, after being broken into small bits, is extracted with acetone until the extracting liquid is practically colourless. The soft resin portion of lac dissolves in acetone during this process, leaving the hard resin portion in an insoluble condition in the extractor. The hard resin complex with urea is now insoluble in alcohol, and in order to make it soluble, it is boiled with water several times, for half an hour each time, with frequent changes of water. After three or four boilings, the hard resin becomes completely free from urea, practically all the urea dissolving in water and being recoverable on evaporation. The hard resin is then dried at room temperature or preferably in a vacuum-drying oven below 42°C.

(2) FRACTIONAL PRECIPITATION METHOD

A known quantity of lac or shellac is dissolved in acetone or acetone-benzene mixture in a vessel provided with a mechanical stirrer. The solution is diluted with a

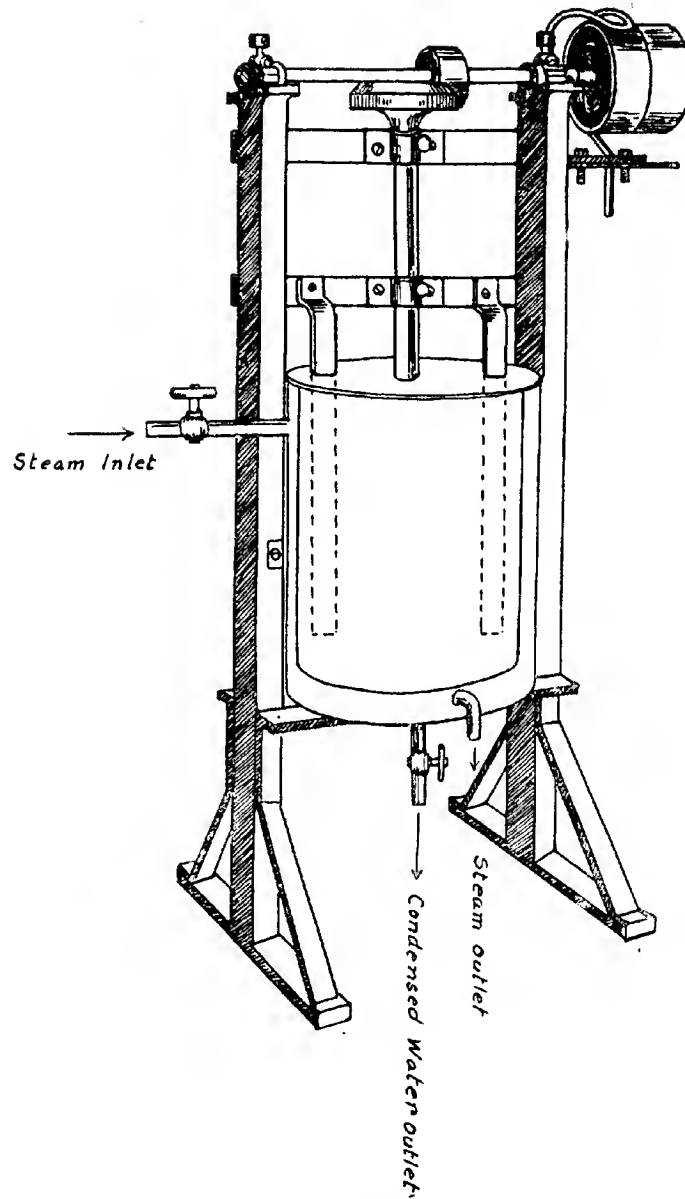


Fig. 8

HARD-LAC EXTRACTOR

further quantity of acetone until the concentration of lac is about 15-20 per cent. The vessel is then externally cooled by ice and the solution kept stirred for about half an hour between 0° and 5°C. The now gelled mass is squeezed through fine muslin to separate the hard and soft resin constituents, the latter being in solution. In order to effect a maximum separation of the soft resin portion, a second washing of the gelled hard resin residue with a further quantity of cold acetone is necessary. The hard resin thus obtained is boiled with water, separated and dried as in the previous method. *Recovery of the solvent from the two processes.*

No special difficulty is experienced in the recovery of the solvent acetone, either from the hard or the soft resin, and any portion of it adhering to the resins does not interfere with their ultimate dissolution in alcohol, a solution which is used in the industry.

(3) BY DIRECT LIQUID EXTRACTION WITH TOLUENE

600 grams of lac are put into a Pyrex flask of 5-litre capacity with a 2-inch diameter neck fitted with a reflux condenser, a thermometer and a mercury seal through which passes the shaft of a collapsible stirrer. Three litres of the extracting solvent, viz., toluene, are introduced through the condenser and the temperature quickly raised to the boiling point. As soon as the resin becomes soft enough to allow free movement of the metal paddles, stirring is commenced. Half an hour is allowed for each extraction after the contents of the flask have been brought to boil. At the end of each boiling period, the flask is detached and the solvent containing the soft resin poured off while still hot. At this stage, a good deal of occluded solvent can be freed by vigorous shaking of the flask. Another 3-litre solvent, preferably pre-heated, is then added and the process repeated five times, i.e., until 15 litres in all of the solvent have been used. At the end of five extractions, the hard resin is poured out of the flask and dried in vacuum. The number of extractions may have to be increased for some very low grades of lac, but good grades may need only four extractions of the type described.

(4) BY FRACTIONAL EXTRACTION WITH WEAK ALKALINE SALTS

The experimental procedure adopted for the extraction of soft resin is based on the following proportions :—

Lac	100 gms.
Water	2,000 c.c.
Sodium carbonate	1.7 gms.
<i>or</i>			
Borax	3.24 gms.

A measured quantity of water is taken in a suitable vessel (provided with a stirrer) (see fig. 8) and the requisite amount of sodium carbonate or borax added. The solution is heated to a temperature of 80°-90°C. when a weighed quantity of lac is added. Stirring is continued until the lac melts and rises to the surface. The extraction takes

15-30 minutes, the time of extraction depending on the quantity of lac used and the rate of heating and stirring. The solution of the soft resin fraction is then decanted (alternatively, the molten hard resin is removed to another vessel). The hard resin thus obtained is then washed free from water-soluble materials. On a small scale this is accomplished by washing three or four times with boiling water. On a large scale, a special stirring device is necessary. Satisfactory results are obtained by grinding the hard resin with water in a ball-mill. The hard resin is then air-dried or vacuum-dried.

It may be mentioned that the special features of this last method of preparing the hard lac resin are (a) less complicated equipment and fairly simple operation, and (b) elimination of the use of solvents, thereby reducing the cost of production considerably.

(5) BY COLD EXTRACTION WITH ETHYL ACETATE

Shellac is finely powdered and dried overnight at about 40-45°C.; it is then charged in a ball-mill with ethyl acetate in the proportion of one pound of the former to 1.5 litres of the latter and the ball-mill is run for about four hours. After this interval, the ethyl acetate which preferentially dissolves out soft resin is decanted out through an ordinary muslin cloth. The ball-mill is recharged with about two-thirds of the original quantity of ethyl acetate and is run for about two hours. The swollen mass of pure resin is now filtered through cloth, pressed very hard to remove the adsorbed ethyl acetate and is spread on floor for drying. The pure resin is obtained in a 60-65 per cent yield with soft resin content of about 2-5 per cent and wax content of about 1-2 per cent.

The ethyl acetate solution of soft resin may be distilled for the recovery of ethyl acetate and the soft resin, the former being used again and again and the latter being utilised for other purposes. Various factors, such as temperature, age of shellac, mechanical treatment during extraction, purity of ethyl acetate, etc., affect the yield and quality of pure resin. Temperature has been found to be a very important factor in the process, higher temperatures giving somewhat less yield but a better product. The most convenient temperature is near about 35°C. The method works more satisfactorily with old or slightly polymerised shellac than with fresh samples. The ethyl acetate used in the first extraction should be very pure (about 98-100 per cent) while for washing in the second operation slightly less pure ethyl acetate may well serve the purpose.

The particular advantages of this method over those already described are :

1. Low working temperature thereby avoiding deterioration of lac by heat-polymerisation.
2. Less risk of fire hazard.
3. Absence of filtering difficulties due to sticky residues.
4. Easy and efficient recovery of solvents.
5. Completeness of separation.

With a view to show the improved properties of the hard lac resin over ordinary shellac, a comparison of their properties is given in the following table :

SHELLAC		HARD LAC RESIN				
		1st method	2nd method	3rd method	4th method	5th method
Softening point (°C.) ...	67	—	87	76	75	81-84
Melting point (°C.) ...	77	—	96	85	87	92-94
Life under heat (min.) ...	58	nil	29	20	12	...
Effect of water immersion (24 hrs.) ...	blushed	no blush	no blush	no blush	no blush	no blush
Effect on copper after baking at 150°C. for 2 hrs. ...	greened and mottled	no greening	no greening	no greening	no greening	no greening
Adhesion and elasticity (baked films) ...	poor	good	good	good	good	good
Scratch hardness kgs. (baked films) ...	5.5	6.5-7.0	6.0	6.5	5.5	6.5-7.0
Acid value ...	71.2	54	56	61.8	56.9	54.0
Yield of soft resin % ...	—	26-28	28-30	26.0	12.18	2.0
Ether soluble resin (%) ...	29.0	3.5	7.10	11.2	22.0	3.0

The hard lac obtained by the methods 1), (2) and (5) contains smaller amounts of ether-soluble soft resin than that produced by the other two methods. The low acid value (54-56) prevents completely the 'greening' on copper. The softening and melting points of the hard lac produced by the second and the fifth methods are higher, which is of great importance in certain industries. Fractionally precipitated hard lac resin from cold acetone solution has comparatively longer 'life under heat,' which is considered to be another advantage.

In view of the special properties, such as higher softening and melting points, and longer 'life under heat,' exhibited by the hard lac resin prepared by the acetone process, the use of this resin is recommended even though the manufacturing cost is a little higher, wherever these qualities are *important*.

Cost of Production

The probable costs for the preparation of hard lac resin on an industrial scale for three of the processes which are comparatively easy of operation are detailed below :

1. By fractional precipitation of hard lac resin in acetone solution.

A plant capable of producing 10.5 maunds of hard lac resin per 24 hours should have the following equipment : --

	Rs.
Building ...	5,000
Refrigerator ...	6,000
Distillation plant ...	4,000
Two filter-presses ...	2,000
Vessels and accessories ...	2,000
Total Rs.	<u>19,000</u>

Cost of manufacturing 10·5 mds. hard lac resin—

	Rs.
15 mds. seedlac at Rs. 20 per md. ...	300-0-0
150 lbs. of acetone lost in process ...	37-8-0
Power costs for refrigeration and distillation	8-0-0
Labour and overhead charges ...	10-0-0
Depreciation, interest, etc. ...	9-8-0
	<hr/>
Total Rs.	365-0-0
¹ Less value of 370 lbs. of soft resin ...	92-8-0
	<hr/>
Total cost Rs.	272-8-0

Net cost per maund of hard lac resin, Rs. 26.

2. *By fractional extraction with weak alkaline salts.*

(A plant capable of producing 12·5 maunds per 24 hrs.)

A. Capital expenditure—

Building ...	5,000
Machinery ...	2,000
Ball-mill ...	2,000
	<hr/>
Total Rs.	9,000

B. Cost of manufacturing 12·5 maunds of hard lac resin—

15 mds. seedlac at Rs. 20 per md. ...	300-0-0
Power costs for heating and stirring ...	8-0-0
Labour and overhead charges ...	10-0-0
Depreciation, interest, etc. ...	5-0-0
Sodium carbonate at Rs. 5 per md. ...	1-4-0
Cost of sulphuric acid for precipitating the soft resin	2-0-0
	<hr/>
Total Rs.	326-4-0
¹ Less value of 196 lbs. of soft resin ...	49-0-0
	<hr/>
Total cost Rs.	277-4-0

Net cost per maund of hard lac resin, Rs. 22-3-0

¹ The soft resin obtained as a by-product can be used directly for oil-varnishes, and after modification, for spirit-varnishes (*vide* Chapters IX and X).

3. BY COLD EXTRACTION WITH ETHYL ACETATE

A. Capital expenditure

					Rs.
Building	5,000
Ball-mills (2)	4,000
Disintegrator	1,000
Distillation Plant	4,000
Filter Presses	2,000
Vessels and accessories	2,000
Total Rs.					18,000

B. Cost of manufacturing 14 maunds of hard resin—

Shellac, 22.5 mds. at Rs. 30 per md.	...	Rs.	675-0-0
Ethyl acetate, 450 lbs. lost in process at 8 as. per lb.	...		225-0-0
Power costs for distillation and ball-milling	...		10-0-0
Labour and overhead charges	...		11-0-0
Depreciation, interest, etc. (15%)	...		9-0-0
			930-0-0
¹ Less cost of 666 lbs. of soft resin recovered at 4 as. per lb.			166-8-0
Total cost Rs.			763-8-0

Net cost per maund of hard resin, Rs. 54-8-6

Hard lac resin has a wide field of application due to its improved properties in many respects over shellac. Some of the more important uses are as follows:

1. Wire enamels for insulation;
2. Inside coatings for tins used for fruit preserves;
3. More heat and water-resistant spirit-varnishes. (It must be added in this connection that films of hard lac resin are less elastic than shellac on air-drying unless suitably plasticised by sextol phthalate, tricresyl phosphate, etc.)
4. Air-drying cements with mineral fillers and rectified spirit.

¹ The soft resin obtained as a by-product can be used directly for oil-varnishes, and after modification, for spirit-varnishes (*vide* Chapters IX and X).

CHAPTER VI

SHELLAC PLASTICS

Numerous articles of everyday use, especially electrical goods, are nowadays made by the plastic moulding of resinous compositions. The most familiar name in this industry is "Bakelite" which is the proprietary name of resin obtained by the combination of phenol and formaldehyde. There are a large number of other plastic materials based upon urea and formaldehyde, glycerine and phthalic anhydride, nitro-cellulose, cellulose acetate, vinyl esters, styrol compounds, acrylates, etc., which are also used in the industry. Plastic goods are manufactured chiefly by three methods, viz., (1) hot moulding which involves the use of moulds which are always kept heated, (2) hot-and-cold moulding, in which the dies are heated up for fusing the composition and cooled down before ejecting the moulded article, and (3) injection-moulding, where the composition is fused in a heated barrel and the fused mass is squirted into cold moulds. The three processes are diagrammatically shown in figure 9.

Bakelite compositions (phenolic) are moulded hot and ejected hot. Shellac is ideally suited for hot-and-cold moulding and is almost universally adopted for the manufacture of gramophone records. (For details of the process the reader is referred to *The Gramophone Record* by H. C. Bryson, and *Uses of Lac*, by H. K. Sen & S. Ranganathan.) Cellulose acetate, polystyrols, etc. are used in injection moulding.

If shellac compositions are to be more widely used for plastic moulding, they should be capable of being used in dies that are always kept hot and should be much more heat resistant than gramophone record compositions. Recent investigations at the Indian Lac Research Institute have shown that shellac modified by formaldehyde and urea could be worked on the hot-moulding technique and shellac filled with jute waste could be injection-moulded, thus making the moulding methods (1) and (3), mentioned above, suitable for shellac compositions. The heat-resistance of hot-moulded shellac articles is about 90° C., which is sufficient for most purposes; by gradual after-baking, the resistance could, however, be raised to 120° C.

HOT-MOULDING

1. *Shellac-Formaldehyde-Urea*

The melting point of shellac can be raised by heat treatment (Nagel, *Metallborse*, Vol. 20, pp. 2133 and 2190). Ranganathan and Aldis (*Bulletin* No. 14 of the Indian Lac Research Institute) observed that many chemicals, especially urea, harden shellac quickly under heat-treatment. Ranganathan (*Bulletin* No. 31 of the Indian Lac Research Institute) prepared shellac moulding powders with urea by the "wet process" but the moulded articles had to be cooled in the dies before ejection. Gardner (*British*

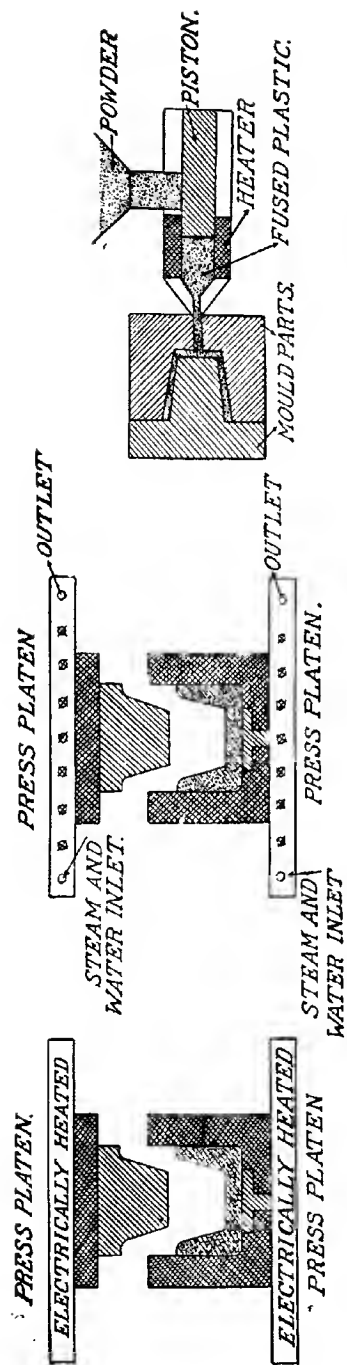


Fig. 9

HOT MOULDING

HOT-AND-COLD MOULDING

INJECTION MOULDING

Plastics, April 1935) prepared shellac moulding powders with oxalic acid, sulphanilic acid, etc., by the "dry process", but in this case too, the dies had to be cooled before ejecting the moulded articles. Venugopalan and Sen (*British Plastics*, April 1939) prepared a modified shellac by treatment with formaldehyde and urea which could be used for compounding moulding powders that may be moulded and taken out of the dies without cooling them.

When shellac is heated with formalin at 120°C., washed in boiling water and tested, it is found to contain 4-6 % formaldehyde, showing that at least a weak combination has taken place. The product is softer than shellac and has longer life under heat.

	Melting point	Life under heat 150° C.
Shellac	78° C.	55 minutes
Shellac-formaldehyde ...	68° C.	90 "

When such a product is reacted with urea and mixed with fillers, etc., a moulding composition is obtained.

The actual procedure to be followed for the preparation of the moulding powder on a commercial scale, and the manufacture of moulded articles are described below and diagrammatically illustrated in Fig. 10.

1. Shellac, formaldehyde, urea, wood flour, calcium stearate, pigment and rectified spirit are digested in a steam-jacketed still, by using steam at 10 lbs. pressure in the jacket for 4 hours, the alcohol being distilled and recovered through a condenser. Towards the end of the distillation, vacuum is employed to effect a more complete recovery (Fig. 11).

The proportions of the different materials employed for the preparation of the moulding powder are :

	Formula A	Formula B	Formula C.
Lac	50.0 lbs.	50.0 lbs.	50.0 lbs.
Formalin (35-40% formaldehyde)	25.0 "	12.5 "	12.5 "
Urea	7.5 "	4.5 "	1.5 "
Calcium stearate	1.25 "	1.25 "	1.25 "
Pigment (brown, black or red)	2.5 "	2.5 "	2.5 "
Wood flour (80-100 mesh)	50.0 "	50.0 "	50.0 "
Rectified spirit	12.5 gals.	12.5 gals.	12.5 "
	(out of which about 10 gallons may be recovered.)		

2. The pasty mass in the still is taken out, and kneaded in a mixing machine of the "Universal" type under vacuum (see Fig. 13), the alcohol vapours being condensed and collected. The dried lumps of the composition are powdered to 60-mesh in a Christy and Norris type of disintegrator (Fig. 14).

3. The powder is finally dried at 90°-95° C. in a vacuum oven for 1-2 hours until the batch sample shows non-blistering and free-flowing properties when moulded

at 120°-150° C. and $\frac{1}{2}$ to 2 minutes in the press (Fig. 12). Thick-walled articles from heavily-built moulds require a lower temperature than thin-walled ones produced in light moulds.

The moulds can be prepared out of mild steel when the moulding is not intricate and when only a few hundreds of articles are required; for complicated mouldings or if these are to be manufactured in thousands, hardened steel moulds are necessary. The polish of the moulds determines the gloss of the moulded surface of the article.

Shellac moulded articles prepared as above can be polished, if necessary, on a buffing wheel or sprayed with lacquers to improve their appearance.

The heat and water resistance of the moulded products are greatly improved by baking them for several hours at 60°C. and then gradually increasing the temperature to 120°C.

The equipment necessary for preparing about 10 cwt. of moulding powder per day of 10 working hours in two charges and the approximate cost sheet for the powder on this scale would be as follows:—

A. Capital Expenditure

			Rs.
Building	5,000
Boiler	4,000
Still (300 gallons), Condenser and Rectifying Column	7,500
Kneading machine (10 gallons)	9,000
Disintegrators (2)	2,000
Drying oven	1,000
Erection charges	1,500
			<hr/>
			30,000
			<hr/>

B. Material Cost

Formula A.

			Rs.	as.	p.
Shellac,	500 lbs. at 6 annas per lb.	...	187	8	0
Formalin,	250 lbs. at 6 " " "	...	93	12	0
Urea,	75 lbs. at 8 " " "	...	37	8	0
Calcium stearate,	12.5 lbs. at 8 annas per lb.	...	6	4	0
Pigment,	25 lbs. at 8 annas per lb.	...	12	8	0
Wood flour (or saw-dust, jute waste or cotton waste)	500 lbs. at 1 anna per lb.	...	31	4	0
Rectified spirit (25 gallons lost in the process at Re. 1 per gallon)		...	25	0	0
			<hr/>		

Material cost for 10 cwt. powder Rs. 393 12 0

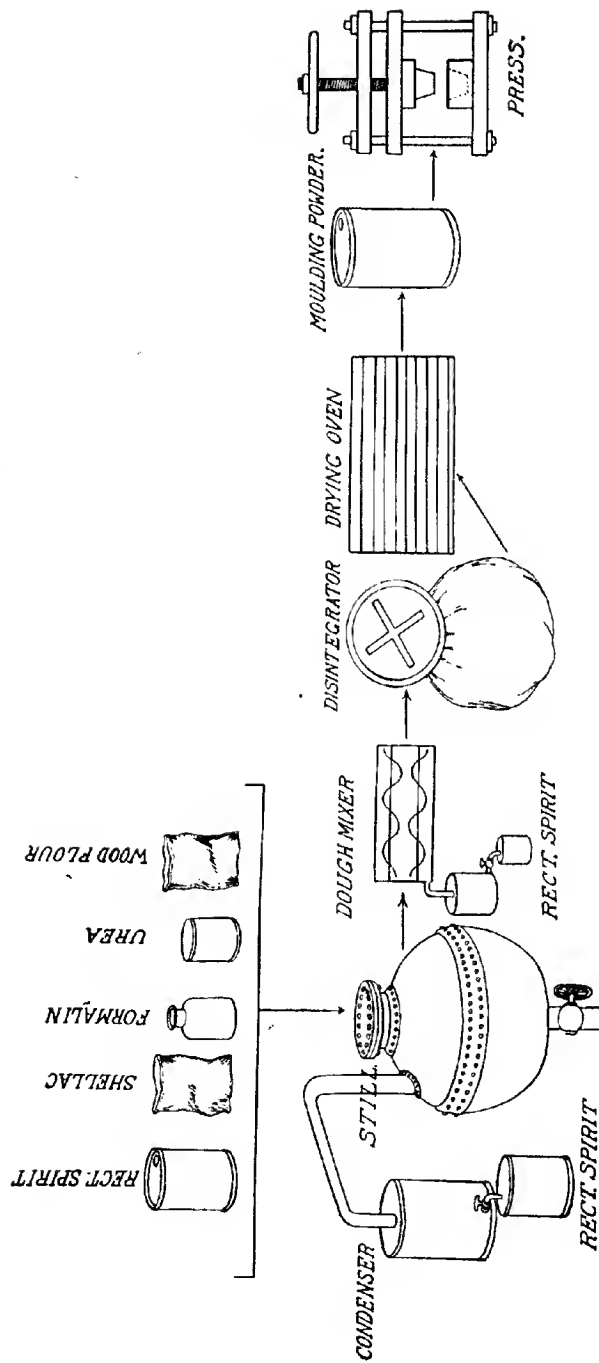


Fig. 10
THE VARIOUS STAGES FROM RAW MATERIALS TO MOULDED ARTICLES

Formula B.

	Rs.	as.	p.
Shellac, 500 lbs. at 6 annas per lb.	... 187	8	0
Formalin, 125 lbs. „ 6 „ „ „	... 46	14	0
Urea, 45 lbs. „ 8 „ „ „	... 22	8	0
Calcium stearate, 12.5 lbs. at 8 annas per lb.	... 6	4	0
Pigment, 25 lbs. at 8 annas per lb.	... 12	8	0
Wood flour (or saw-dust, jute waste or cotton waste) 500 lbs. at 1 anna per lb.	... 31	4	0
Rectified spirit (25 gallons lost in the process at Re. 1 per gallon)	... 25	0	0

Material cost for 10 cwt. powder Rs. 331 14 0

Formula C.

	Rs.	as.	p.
Shellac, 500 lbs. at 6 annas per lb.	... 187	8	0
Formalin, 125 lbs. „ 6 „ „ „	... 46	14	0
Urea, 15 lbs. „ 8 „ „ „	... 7	8	0
Calcium stearate, 12.5 lbs. at 8 annas per lb.	... 6	4	0
Pigment, 25 lbs. at 8 annas per lb.	... 12	8	0
Wood flour (or saw-dust, jute waste or cotton waste), 500 lbs. at 1 anna per lb.	... 31	4	0
Rectified spirit (25 gallons lost in the process) at Re. 1 per gallon	... 25	0	0

Material cost for 10 cwt. powder Rs. 316 14 0

1. Process charges including depreciation, interest, etc.

	Rs.	as.	p.
Chemist Manager @ Rs. 150 p. m.	... 5	0	0
Mechanic @ Rs. 90 p. m.	... 3	0	0
Boiler man @ Rs. 30 p. m.	... 1	0	0
Labourers (10) @ 12 annas per day	... 7	8	0
Steam charges (10 mds. coal @ 8 annas per md.)	... 5	0	0
Electricity charges (32 KWH @ 1 anna)	... 2	0	0
Depreciation on building and machinery @ 10% of capital	10	0	0
Interest on capital @ 5 %	... 5	0	0
Office expenses	... 5	0	0
Total processing charges	... 43	8	0

The cost of 10 cwt. of moulding powder, including the processing charges, would therefore work out to Rs. 437-4-0, for the formula A or about 6·2 annas per pound. Corresponding cost per pound of the powders prepared according to formulae B and C would be about 5·3 annas and 5·1 annas respectively. If instead of shellac, seedlac or lac recovered from by-products like *kiri*, etc., is used, the price would be reduced still further.

As regards the equipment, the still should be lined with tinned copper and should be jacketed for heating by steam. The manhole at the top for charging and the discharge hole at the bottom should be about 12 inches in diameter for easy working. A centrally mounted stirrer with stoutly constructed fins of stainless steel is necessary for efficient mixing while the digestion is proceeding. The kneader-mixer should be totally enclosed with arrangements for external steam-heating and solvent recovery, under vacuum, to minimise loss of alcohol. Disintegrators of the grinding discs type are unsuitable and the beater type of machine (Carter or Christy-Norris) should be used.

Except the shellac and rectified spirit, all other materials have now to be imported. Wood flour can be prepared in this country if there is a demand, but waste cotton or jute can be substituted for wood flour. Processes for the manufacture of calcium stearate, urea, and formaldehyde on a small scale have been worked out at the Institute, details of which could be supplied on request. Indigenous pigments could be easily substituted for the now imported ones.

About 5000 lbs. of moulding powders have been prepared at the Institute and supplied to moulding companies principally for the manufacture of switches and ceiling roses, and 10,000 insulation boards for fan regulators have been pressed and sold to several firms in India.

As an example of commercial plastic goods production, the manufacture of 5 amp. switches may be considered and an estimated cost sheet for the same is given below :—

<i>A. Capital Expenditure</i>			Rs.
Building	4,000
Hydraulic presses (2) (60-ton semi-automatic)	10,000
Two switch moulds (4-cavity)	4,000
Mould for dolly (8-impression)	1,000
Air compressor, tools, etc.	1,000
			<hr/>
			20,000
			<hr/>

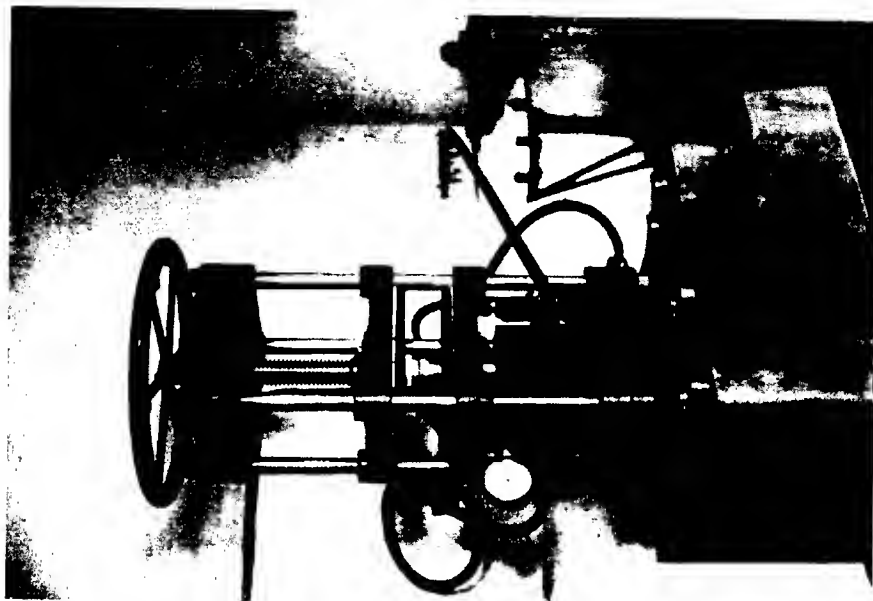


Fig. 12
HYDRAULIC PRESS

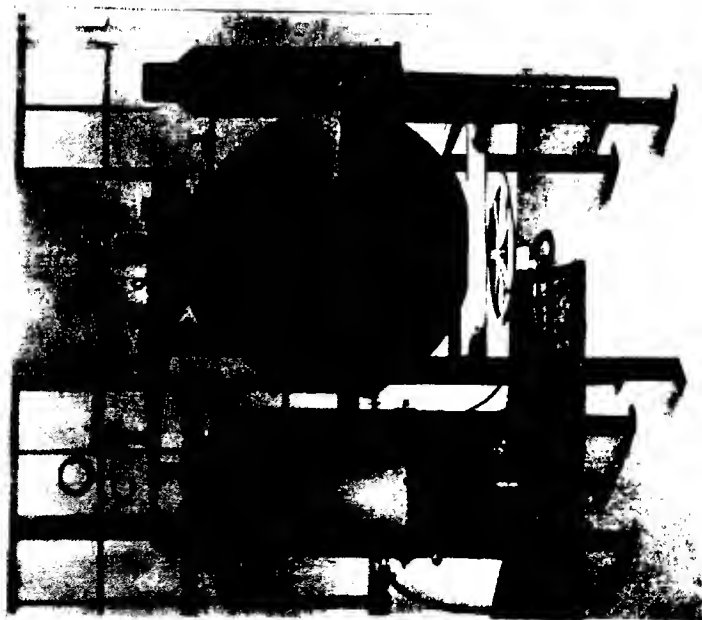


Fig. 11
STEAM-JACKETED DIGESTOR
(Ellis, *The Chemistry of Synthetic Resins*)

B. Cost of manufacturing 120 doz. electric switches per day of 24 hours in 8 shifts

	Rs.	as.	p.
Moulding powder, 120 lbs. @ 5.3 annas per lb. ...	39	12	0
Electric Power, 160 KWH @ 1 anna per KWH ...	10	0	0
Labourers (15) @ 12 annas each ...	11	4	0
Supervision (3 men @ Rs. 2 each) ...	6	0	0
Cost of metal parts @ 12 annas per doz. sets ...	90	0	0
Buffing or spraying and assembling @ 3 annas per doz. ...	22	8	0
Repairs and sundry expenses ...	2	0	0
Management and Office expenses ...	10	0	0
Depreciation @ 10% on capital (Rs. 20,000) ...	6	10	8
Interest @ 5% ...	3	5	4
Incidental expenses ...	10	0	0
Cost of 120 doz. switches ...	211	8	0
or Rs. 1-12-2 per doz.			

The manufacture of the moulding powder described above requires denatured spirit as an essential commodity although most of it is recovered in the process. The loss of spirit which on an average is about 15% of the quantity used could be overlooked at a time when the spirit cost only 8 annas a gallon. This, however, is no longer the case as from 8 annas per gallon it has risen to Rs. 4 even at the centres of production. Accordingly, to mitigate the high cost of the shellac moulding powder, the avoidance of the use of spirit and the costly equipment in the shape of still, rectifying column, etc., was attempted at the Institute by having recourse to hot-roller mixing, and a few compositions have been formulated requiring less or no alcohol and very much reduced quantities of imported costly chemicals like urea and formalin.

2. Hot-roller mixed compositions

The preparation of the moulding powder by hot-roller mixing which has formed the subject of a patent (Indian Patent No. 29329) is described below:

(i) Lac (extracted from *Kiri*) is heated with filler and modifying agents in the following proportions at 100-110°C. for 2 hours in a kneader mixer.

Lac ...	100	lbs.
Saw-dust 80-90 mesh ...	90	"
Formalin (40%) ...	20	"
Urea or Thiourea ...	3	"
Stearic acid ...	1.25	"
Calcium oxide ...	1	lb.
Pigment ...	2.5	lbs.
Water ...	10	gals. ¹

¹ A slight variation in the above formula is to substitute denatured spirit for part of the water, and recovering most of the spirit during the mixing and kneading.

The mass is then mixed between heavy steam-heated rollers (100–110°C.) moving towards each other at different speeds.

(ii) If waste pulp from paper mills or waste jute is used as filler the materials in the following proportions are mixed together and left to stand overnight before hot-roller mixing is done :

Lac	...	28	lbs.
Formalin (40%)	...	7	"
Calcium oxide	...	0.28	lb.
Urea or Thiourea	...	0.84	"
Pulp or jute waste	...	22.25	lbs.
Stearic acid	...	1	lb.
Pigment	...	1	"
Water	...	28	lbs.

Instead of 28 lbs. of water, a mixture of 17 lbs. of denatured spirit and 11 lbs. of water gives a better moulding powder.

The wet mixture is rolled between hot-rollers with less pressure between them while using jute so as to avoid cutting the fibres too short. When all the moisture has been removed and the material is uniformly well-fused into a sheet it is taken out, powdered and dried. The impact strength of the moulded articles containing pulp or jute waste is 5–6 cm. kg./sq. cm.

(iii) A composition not containing urea or formalin and yet suitable for hot moulding is given below :—

Lac	...	100	lbs.
Water	...	20	gals.
Liquor ammonia (0.88)	...	6.6	lbs.
Slaked lime	...	1.7	"
Stearic acid	...	2	"
Saw dust or pulp wastes	...	100	"
Pigment	...	3	"

The lac is soaked in dilute ammonia for half-an-hour, after which the other materials are added and mixed between hot-rollers. The homogeneous sheet of the composition is powdered, dried and used for moulding. The moulded articles have an impact strength of 3–4 cm. kg./sq. cm.

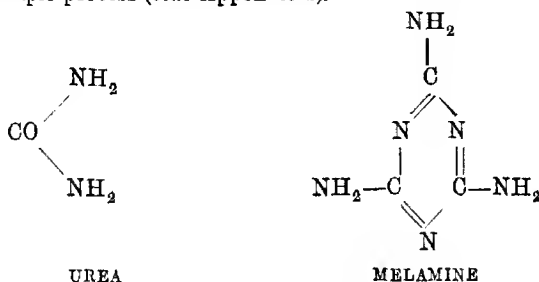
In all the above compositions prepared by either the wet process or the dry hot-roller mixing process, incorporation of 5–10 per cent plasticising agents like phenolic fractions from coal tar, ester gum, cashew shell liquid, etc., has been found to improve the flow of the moulding powder and finish of the moulded articles.

Below are given a few of the typical properties of shellac compositions with organic fillers.

Compression Moulding Temperature °C.	...	130-150
Compression Moulding Pressure, lbs. per sq. inch	...	2240-4480
Compression ratio	...	2-3
Specific Gravity	...	1.3
Impact strength, cm. kg./sq. cm.	...	3-6
Thermal Conductivity, 10^{-3} cal. per sec. per sq. cm./°C. per cm.	...	2.3-4.5
Resistance to Heat, °C. Marten's	...	70-90
Tendency to cold flow	...	none
Volume Resistivity, ohms-cm. (50% relative humidity & 25°C.)	...	$(3-5) \times 10^{14}$
Surface Resistivity, ohms-cm. (50% relative humidity & 25°C.)	...	$(1-3) \times 10^{15}$
Dielectric strength, short-time, volts per mil. $\frac{1}{8}$ in. thickness	...	250-450
Dielectric Constant 50 cycles	...	3.5-5.0
Dielectric Constant 10^3 cycles	...	3.5-4.5
Dielectric Constant 10^6 cycles	...	3.5-4.3
Power Factor 50 cycles	...	0.01-0.03
Power Factor 10^3 cycles	...	0.008-0.02
Power Factor 10^6 cycles	...	0.005-0.015
Water Absorption 24 hours	...	1-1.5%
Effect of Age	...	none
Effect of sunlight	...	none
Effect of weak acids	...	none
Effect of strong acids	...	deteriorates
Effect of weak alkalies	...	"
Effect of strong alkalies	...	"
Effect of organic solvents	...	Resists aliphatic hydrocarbons and oils. Soluble in ketones and esters. Swells in aromatic hydro- carbon solvents.
Effect of metal inserts	...	Inert
Machining qualities	...	Good
Clarity	...	Opaque
Colour Possibilities	...	Limited, pastel excluded.

3. SHELLAC-FORMALDEHYDE-MELAMINE

Attempts to further harden shellac by accelerators other than urea have led to the use of melamine which has been found to confer improved properties. Melamine is now a commercial product as it is readily manufactured from technical calcium cyanamide through a simple process (*vide* Appendices).



The equipment necessary and the general process for the production of shellac-formaldehyde-melamine powder are exactly analogous to those of the shellac-formaldehyde-urea moulding powder, but the quantities of formaldehyde and melamine to be used are less. The best proportions have been found to be as follows:—

Shellac	100 lbs.
Formalin	20 lbs.
Melamine	5 lbs.
Calcium stearate	2.5 lbs.
Pigment	2.5 lbs.
Wood flour (or other fibrous filler)			100 lbs.

The powder can be used for moulding articles at 140°–145°C. and these ejected from the moulds at the same temperature. Below is given the approximate cost-sheet of materials for the production of shellac-formaldehyde-melamine moulding powder.

		Rs.	A.	P.
Shellac, 100 lbs. at As. 6 per lb.	...	37	8	0
Formalin, 20 lbs. at As. 6 per lb.	...	7	8	0
Melamine, 5 lbs. @ Rs. 2/8/- per lb.	...	12	8	0
Calcium stearate, 2.5 lbs. @ As. 8 per lb.	...	1	4	0
Pigment, 2.5 lbs. @ As. 8 per lb.	...	1	4	0
Filler, 100 lbs. @ As. 1 per lb.	...	6	4	0
Rectified spirit lost, 5 gals.	...	5	0	0
Total cost of materials for 200 to 210 lbs.	...	71	4	0

The same resin in the original alcoholic solution may also be used as a short-baking varnish of high heat and water resistance. By impregnating jute and cotton fabrics with this varnish, drying and pressing hot under the hydraulic press, laminated pieces of remarkable hardness can be produced.

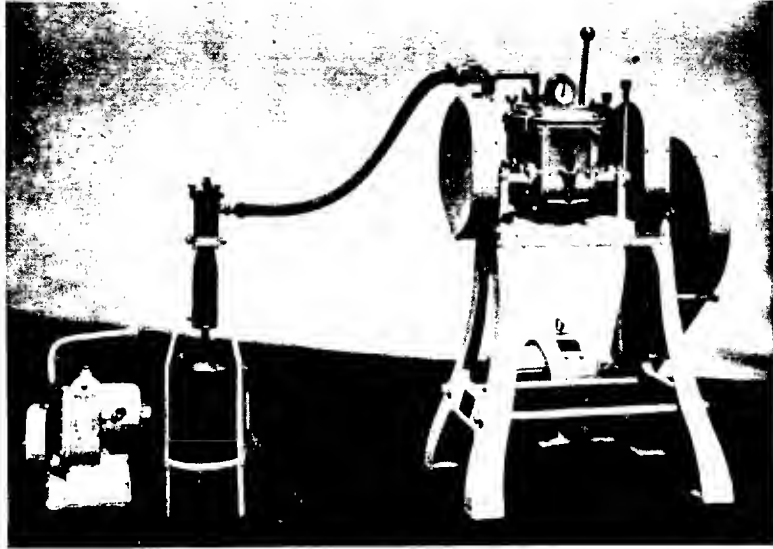


Fig. 13
"UNIVERSAL" MIXING MACHINE

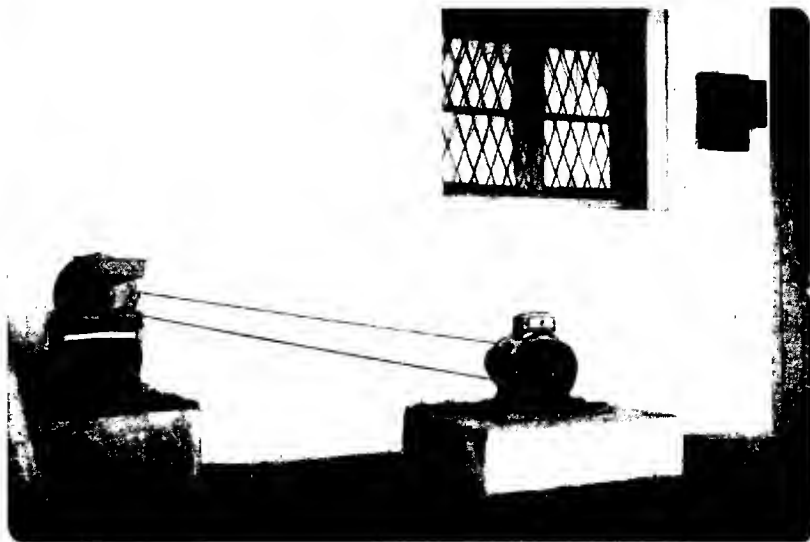


Fig. 14
CHRISTY AND NORRIS DISINTEGRATOR

4. SHELLAC-COALTAR

Experiments have been conducted with a view to finding cheaper materials which can replace either partly or wholly the chemicals used in the foregoing compositions. Moreover, the need for very cheap moulded slabs either for insulation or for constructional purposes became apparent from trade enquiries. To meet these and other requirements, coaltar, which contains both phenolic bodies and unsaturated neutral oils, was considered a suitable addition material to the usual composition. Experiments on these lines have shown that as much as 25-30 per cent of dry tar could be compounded with shellac, while at the same time a considerable reduction in the quantities of urea and formaldehyde could be effected. A typical experiment was conducted as below:

100 parts of lac were mixed with 10 parts of para-formaldehyde and heated for one hour at 120°C., at the end of which 25 parts of dry tar or crude carbolic acid were added and further heated for $\frac{1}{2}$ hour at the same temperature. The resin so formed was then dissolved in 200 parts of rectified spirit and to the solution 6 parts of urea were added and the whole refluxed for 2 hours. The resin solution so obtained was then mixed with 100 parts of wood flour and 1.5 parts of calcium stearate and thoroughly kneaded and air-dried. The powder was then crushed in a disintegrator to 30-mesh and dried at 90°-95° C. for 3-4 hours. It could be moulded at 140°-145° C. under 1 to 1.5 tons pressure per sq. inch. The moulded articles develop considerable hardness and heat and water resistance on baking at 120°C. Only black colour is possible in the mouldings, unless suitably lacquered.

The alcoholic solution of this resin can be used as a cheap baking varnish of considerable water- and scratch-resistance.

5. SHELLAC-CASEIN

Although, under normal conditions of the world-market, shellac-formaldehyde urea moulding powders can be produced definitely cheaper than bakelite, the difficulty of importing urea and formaldehyde into this country, has made this impossible. Until such time as these two chemicals are manufactured in India or are regularly available from foreign sources, the introduction in India of these powders would no doubt be greatly handicapped. For this reason, investigations were continued to develop a moulding powder entirely independent of foreign chemicals. Milk casein, of which about 8,000 cwt. are exported annually, was thought of as a suitable material, having amino and amido characteristics for combination with shellac. This has yielded, as expected, good moulding powders with better fluidity but somewhat less water resistance. The water resistance can, however, be improved by coating the moulded articles with shellac-nitrocellulose lacquers. On subsequent baking, the moulded products leave little to be desired. The strength of the articles made from a shellac-casein composition appears to be slightly less than that of those

from shellac-formaldehyde-urea powders, but this is within a range which does not preclude their use for most purposes. A typical process for preparing this powder is given below :

450 gms. of shellac are dissolved in 1000 c.c. of water containing 50 c.c. of liquor ammonia (sp. gr. 0.88). This solution is mixed hot with an ammoniacal casein solution prepared by dissolving 56 gms. of casein in 250 c.c. of water containing 5-8 c.c. of liquor ammonia. The above resin solution is mixed with 5 gms. of lime dispersed in a little water, 450 gms. of wood flour, 12.5 gms. of calcium stearate and 20-25 gms. of pigments, and the pasty mass so obtained is thoroughly kneaded. It is then air-dried and powdered to 60-mesh. The powder is finally dried for 4-5 hours at 95°-97°C., after which it is ready for moulding. The test bars moulded out of this powder have an impact strength of 3.5 cm. kg./sq. cm. and a heat resistance of 93°C. Subsequent baking improves the heat resistance to over 110°C. without loss of strength. Articles moulded at 135°-140°C. under $1\frac{1}{2}$ -1 $\frac{3}{4}$ tons per sq. inch pressure can be ejected from the press without distortion, and when subsequently polished on the flannel buff, take excellent gloss.

The shellac-casein composition is considerably cheaper than shellac-urea-formaldehyde moulding powder, the ingredients required being cheap and obtainable in desired quantities in the country. Besides, the method of manufacture of this powder is extremely simple, practically no special equipment being necessary. Casein from vegetable products and other proteins from soya bean, tamarind seeds, etc., serve the purpose equally well. In fact, a cheapest variety of oil-cake (*Karanj*—*Pongamia glabra*) available in this locality, when crushed and extracted with ammonia, gives an emulsion which can be directly used with ammoniacal shellac to obtain a moulding powder.

6. SHELLAC-CASHEW NUT SHELL OIL

In view of the fact that large quantities of cashew shell liquid obtained as a by-product during the roasting process employed for the separation of the kernel are available in this country, several investigations were conducted with the object of utilising this oil in combination with shellac for both moulding and varnish purposes. Improvements noted in the varnish properties obtained by the combination of shellac and this oil are recorded in a separate chapter, and in this attention is confined to the properties of the moulding powder obtained as the result of the lac-cashew nut oil combination.

It has been found that incorporation of 15-25% of cashew nut shell oil either as such or previously polymerised with formaldehyde in presence of ammonia as catalyst (in both the 'wet' and 'roller mixing' processes of making the composition in presence of suitable hardeners) improves considerably the flow and finish of the moulding compositions. It has also been observed that the water resistance of the moulded articles increases considerably. In a typical composition water absorption

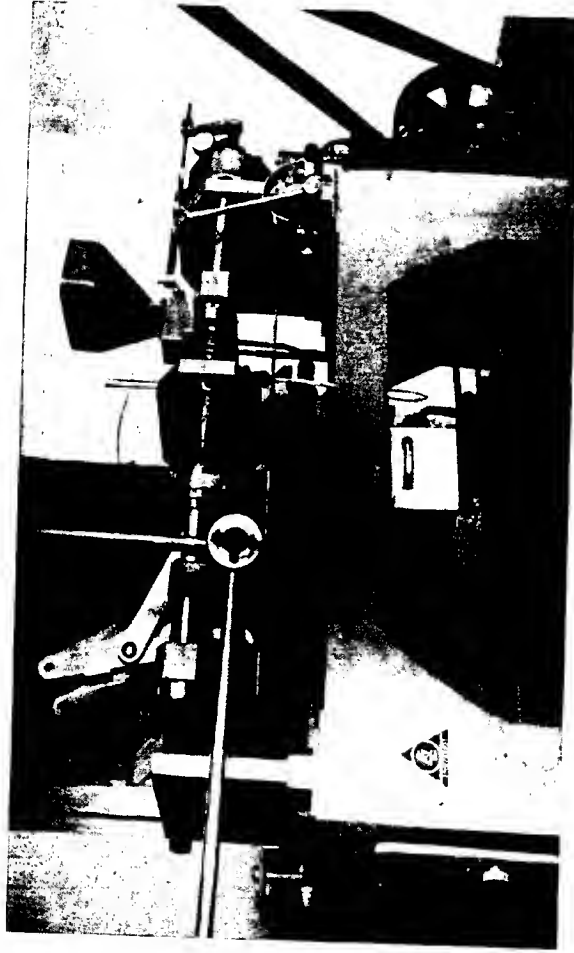


Fig. 15
INJECTION MOULDING PRESS

of about 0.4-0.5% in 24 hours has been found as against 1-1.5% for the control. The heat resistance and impact strength of the compositions are, however, not increased and work on the improvement of these two qualities is in progress.

Injection Moulding

Injection moulding may be defined, in the words of Isermann Buchholz as 'the process which comprises heating the material to be moulded until the same acquires flowing characteristics, then forcing the said material directly from the point of heating through a relatively restricted passage under pressure to increase its flowing characteristics and in this condition forcing it into the mould and cooling the mould during the introduction thereto of the fluid material'. The essential parts of the injection moulding press (Fig. 15), therefore, are the hopper for feeding the powder, the pressure head (mechanical, hydraulic or air), the working piston, the electrically heated barrel, the nozzle and the mould in two parts complete with the locking device.

The moulding material in granular form is fed into the hopper at the extreme right, wherefrom it is automatically sucked into the barrel by the movement of the piston. Here it is heated up by external and internal electric heating elements until it becomes plastic. The mould is now closed and the pressure valve released. The pressure ram is driven forward 'like a shell fired from a gun' resulting in the injection of the fluid material with great rapidity into the mould where it is instantly chilled.

With machines working on this principle (which are of the utmost simplicity), it is possible to produce two to five impressions per minute depending on the thickness of the desired article and the efficiency of the water-cooling arrangement. With a multi-impression mould the output may be increased several times. In the fully automatic and semi-automatic machines (vide *British Plastics*, April, 1934, p. 504) there are motor controls for the closing and opening of the mould as well as for fixing the mould on to the nozzle. Two time switches control the time of pressing as well as the time necessary for heating the material newly introduced into the injection cylinder. Another switch serves for instantly changing the working from fully automatic to semi-automatic or *vice versa*. Such a machine can be made to stamp about five impressions a minute for ordinary, thin-walled articles. The efficiency and speed of working of such a press will be appreciated when it is pointed out that when fitted with a twelve-impression mould for tooth-paste tube caps, after feeding the material into the hopper, one can collect at the rate of nearly 3,600 pieces per hour without any other manipulation.

Injection moulding machines are marketed with different capacities ranging from two to nearly sixteen ounces. There are also machines available with two or four injection units in the same press and the capacity of some of these is as high as 36 oz. per shot, so that they may be used for moulding particularly large pieces like window frames and steering wheels for automobiles, etc. Most of these large-capacity

machines are also provided with force-feeds from the hopper providing positive delivery of the necessary quantity of moulding material into the barrel without break.

The thermoplastic materials most commonly used for injection moulding are those produced from cellulose acetate and styrol resins, and recently benzyl cellulose. These are marketed under several trade names, e.g., trolit, cellomold, tenite, trolitul, etc., to mention only a few.

The difficulties in using shellac for injection moulding are its (i) low softening point (ii) brittleness and (iii) slow thermohardening.

Brittleness is readily overcome by using fillers. Mineral fillers alone like barytes, kaolin, slate dust, etc., result in articles which are unnecessarily heavy and still brittle.

Fibrous fillers improve the shock resistance but the appearance leaves much to be desired. A combination of fibrous and mineral fillers gives satisfactory results. Waste jute and china clay give the best results.

The addition of urea imparts certain desirable degree of hardness and heat resistance to the moulded articles, but this is beset with a practical difficulty in that if the moulding operation is interrupted for more than a few minutes, the composition tends to harden in the barrel of the injection moulding press thereby losing its fluidity. To avoid this trouble other accelerators were tried and as a result of a series of experiments, the following alkaline accelerators in the proportion mentioned against each were found to be suitable.

Guanidine carbonate	...	5%	on the wt. of lac
Guanyl urea	...	5%	" " " " "
Liquor Ammonia (0.88)	...	3%	" " " " "

When shellac together with the proper amount of fillers is well-kneaded with an aqueous solution of any one of these bases in the respective proportions, dried in the air, run through hot rollers, crushed and cured at 85°-90°C. for 1½ hours, the powder obtained thereby flows at 135°-150°C. which is 45°-50°C. higher than the corresponding temperature for ordinary shellac powder. The articles resist a temperature of 65°-70°C. without distortion or blistering and are sufficiently glossy and strong. The impact strength of moulding composition is 4.2 to 4.4 cm. kg/cm.²

Below are given two typical injection moulding compositions found to be easily workable for the large-scale production of a number of useful articles.

(1)	Lac (shellac) 30-mesh	...	100	lbs.
	Jute waste 12-18-mesh	...	66.6	"
	Kaolin	...	66.6	"
	Stearic acid	...	3.0	"
	Pigment	...	10.0	"
(2)	Lac (shellac) 30-mesh	...	100	"
	Jute waste 12-18-mesh	...	66.6	"
	Kaolin	...	33.3	"

Calcium stearate	...	5.0	lbs.
Pigment	...	10.0	"
Liquor Ammonia	...	3.0	"
Water	...	25	gals.

In both the cases the materials are first mixed at the ordinary temperature and then between steam-heated rollers to obtain a homogeneous mixture. The material which is obtained in the form of a sheet from the roller is allowed to cool and then powdered and dried for 1-1½ hours at 90°C. It is then used for moulding.

Powder No. 2 containing ammonia produces articles which are more heat-resistant, but less glossy than those prepared in the absence of ammonia.

It will be noted that all the ingredients mentioned in the above compositions are readily available in India in sufficient quantities and at cheap rates. The process of manufacture is simple and requires only the following special equipment :—

- (i) A boiler
- (ii) A hot roller mixer (steam-heated)
- (iii) A Christy & Norris disintegrator
- (iv) A shelf drying oven (steam-heated)
- (v) A grinder

With a hand-worked Eckert & Ziegler machine as shown in Fig. 15, the following was found to be the rate of production for a few typical articles :—

<i>Articles</i>	<i>Mould</i>	<i>Rate per hour</i>
Bottle caps	4-impression	360-400
Kit-kat switch bases	Single "	70-75
" " covers	Single "	150-180
" " knobs	4-impression	300-360

The rate of production largely depends on the design of the mould and the thickness and complexity of the articles. About 60,000 containers (2 oz. capacity) and 10,000 electric switches (3 amp. kit-kat type) were made at the Institute and supplied, thus demonstrating the workability of the powder and the process on a commercial scale.

It must be noted that no claim is made that shellac injection moulding powder is comparable to the cellulose or styrol base powders. So far attempts to prepare transparent moulding powders from shellac have not been successful and they can never be obtained colourless. But for inexpensive household articles it will have an advantage over the imported powders in view of its cheapness, the powder costing only six annas a lb. compared with Re. 1-8 to Rs. 2 for other powders even at pre-war rates. It will be of interest to note that even in foreign countries, shellac injection moulding powders are already being largely used for the production of toys and the like.

The cost of equipment and production are as follows :—

<i>Capital Expenditure.</i>	Rs.
Building	2,500
Injection moulding machine	8,000
Boiler	1,000
Hot roller mixer	800
Shelf drying oven	500
Disintegrator	600
Equipment	1,000
Miscellaneous	600
	<u>Rs. 15,000</u>

<i>Moulding powder</i>	Rs.	a.	p.
Lac (56 lbs. @ 6 as. per lb.)	21	0	0
Jute 38 lbs. (including grinding)	3	9	0
Kaolin (19 lbs.)	0	9	6
Pigment (2½ lbs. @ 8 as. per lb.)	1	6	0
Calcium Stearate (1½ lbs. @ 8 as. per lb.)	0	12	0
Ammonia (1½ lbs. @ 6 as. per lb.)	0	10	6
Labour	1	4	0
Supervision	3	0	0
Depreciation and interest	3	8	0
Miscellaneous	5	10	6

Total for 1 cwt. 41 5 6

Therefore, cost per lb. 0 5 10

<i>Kit-kat Switches, 2 gross per day, working 8 hours</i>	Rs.	a.	p.
(Powder 24 lbs. @ 6 as. per lb.)	9	0	0
(Power 30 units)	3	4	6
Labour	1	0	0
Supervision	3	0	0
Depreciation and interest	5	0	0
	<u>21</u>	<u>4</u>	<u>6</u>

Therefore, cost per doz. 0 14 2

<i>Bottle Caps: 15 gross working 8 hours</i>	Rs.	a.	p.
(Powder 20 lbs. @ 6 as. per lb.)	7	8	0
(Power 30 units)	3	4	6
Labour	1	0	0
Supervision	3	0	0
Depreciation and interest	5	0	0

Total for 15 gross. 19 12 6

Therefore, cost per gross. 1 5 1

FILLERS

In the plastic industry, specially prepared wood flour is universally used as filler in the formulation of the moulding powders, in order to impart good mechanical strength to the moulded articles. In the shellac moulding compositions described above, saw-dust, jute waste or paper pulp is recommended as alternative materials to the specially prepared wood flour which is generally made by crushing waste wood chips in stone grinders or attrition grinders so that the particle size is decreased still maintaining fibrous texture instead of being cut across. In order to make saw dust suitable as filler it should be treated as follows: Saw-dust, collected in mills and carpenters' workshops, is first sieved through a 5-10-mesh sieve and then levigated in water to remove inorganic impurities that might be accidentally present, and dried. It is then ground in a Christy-Norris type of disintegrator, passed through a 80-mesh sieve and then levigated again more than once, if necessary, and finally dried. Saw-dust from all the ordinary timbers which have an ash content of about 2-3% can be used after being cleaned by washing and grinding as above, but Haldu (*Adena cordifolia*) and Gangwa (*Excocaria agallocha*) have been found to yield stronger moulded articles. Teak dust after similar treatment has a special applicability as filler, as it reduces water absorption in the moulded articles. Comparative data on imported wood flour and clean saw-dust are given below:

		<i>Imported wood flour</i>	<i>Saw-dust</i>
Acetone extract	...	Below 0.2 per cent	3-4 per cent
Ash	...	Below 1.0 " "	4-6 " "
Bulk (i. e. volume occupied by 25 grams when poured into a 200 c. c. cylinder and gently tapped)	...	120-140 c.c.	100 c.c.
Fineness generally used	...	80-120-mesh	80-100-mesh
Impact strength of moulded bars under otherwise equal conditions	...	6-7 cm. kg./cm. ²	5-6 cm. kg./cm. ²

It would appear from above, that articles moulded from compositions containing saw-dust as filler are somewhat weaker in strength, which is only to be expected from the difference in the fibrous structure of saw-dust and wood flour. This can be known by examining them under the microscope. Jute-waste has also been examined as a filler. This is cheap but requires cleaning from grease and bits of stumps associated with it. The fibres are ground in a disintegrator so that fibre lengths of less than one inch are obtained. The general effect of admixing such a jute filler with the shellac moulding composition for compression moulding does not appear to be as satisfactory as with wood-flour or clean saw-dust. The use of jute fibre, however, for

injection moulding is quite satisfactory and 60,000 anti-mosquito cream containers have been supplied to the military with ground jute fibre as filler by the injection moulding process and these are reported to be satisfactory.

Clean paper pulp and even pure alfa-cellulose are generally used as fillers for urea-formaldehyde moulding powders particularly as these confer better shock-resisting properties on the mouldings than wood flour. Shellac moulding powders with paper pulp as filler yield strong, glossy articles but the cost is increased thereby. Cotton linters could also be used as filler, but the powders are too light and have to be tableted before use, or deeper moulds have to be employed.

CHAPTER VII

ADHESIVES AND CEMENTS

Apart from the more important applications of shellac in the electrical, plastic and varnish industries, its use in the manufacture of adhesive and cementing materials is not inconsiderable. The excellence of adhesion between shellac and such varying materials as mica, metals, marble, etc., is well-known and it is, therefore, used as a cement or adhesive in a variety of applications. Its utilisation in the manufacture of laminated fabrics, its use as a cement between glass and metal parts, and for protecting photographic negatives, are but a few of the typical examples. In all these applications, the cement or adhesive is dark-coloured or opaque and as such its use in articles requiring transparency and clearness is limited. With a view to overcome these limitations and thereby increase the application of shellac in this field, attempts were made to prepare a water-white, transparent adhesive, starting from one of the major constituents of shellac. A brief description of the preparation of this new adhesive is given below :

500 gms. of aleuritic acid (*vide appendix*) are condensed with 120-150 grams of phthalic anhydride at 180°-190°C. for 20-30 minutes. The addition of a small amount of glycerine (5-10 per cent) facilitates easy condensation and prevents the phthalic anhydride from volatilising during the reaction. The condensed mass is heated with 250 c. c. of formaldehyde (40 per cent solution) for 2 hours. The resulting product is dissolved in butyl alcohol and 75-100 grams of urea are added slowly to prevent a quick reaction. The whole is further heated with a reflux condenser for 2 hours and then vacuum-distilled to remove the solvent and water. The resulting clear, thick and viscous compound may be used as an adhesive, either as such or in solution in a mixture of alcohol and benzene or alcohol and toluene.

Cost of Preparing the Adhesive			Rs.		
A. Capital Expenditure	1,000		
B. Materials—			Rs.	A.	P.
Aleuritic acid, 10 lbs. @ As. 8 per lb.	5	0	0
Phthalic anhydride, 2.5 lbs.	1	2	0
Formalin, 5 lbs. @ As. 6 per lb.	1	14	0
Glycerine, 1 lb. @ As. 8 per lb.	0	8	0
Urea, 2 lbs. @ As. 8 per lb.	1	0	0
N-Butyl alcohol, 10 lbs. @ As. 7 per lb.	4	6	0
Labour, overhead charges, depreciation, interest, etc.	3	10	0
		Total Rs.	17	8	0
Less cost of 8 lbs. butyl alcohol recovered	3	8	0
		Cost of 20 lbs. of adhesive Rs.	14	0	0
		or cost per pound As. 11-2			

The adhesive may be made heat and water-resistant by baking the articles bonded with it at 80°-90°C. for 15 minutes. Owing to its property of water-clearness and transparency, its use in the manufacture of safety glass, laminated articles, etc., is suggested.

Various new uses of shellac or modified shellac adhesives are being investigated at the laboratories of Messrs. Metropolitan Vickers Electrical Company in London and the Indian Lac Research Institute at Ranchi. One such application which has given definitely promising results is in the use of shellac modified with phthalic anhydride as an adhesive for the bonding of paper in the manufacture of 'Traffolite' decorative boards for wall-panels, table-tops, etc. The use of greased paper coated with shellac varnish treated with phthalic anhydride gave better results than the phenolic resin varnish used at present as the adhesive.

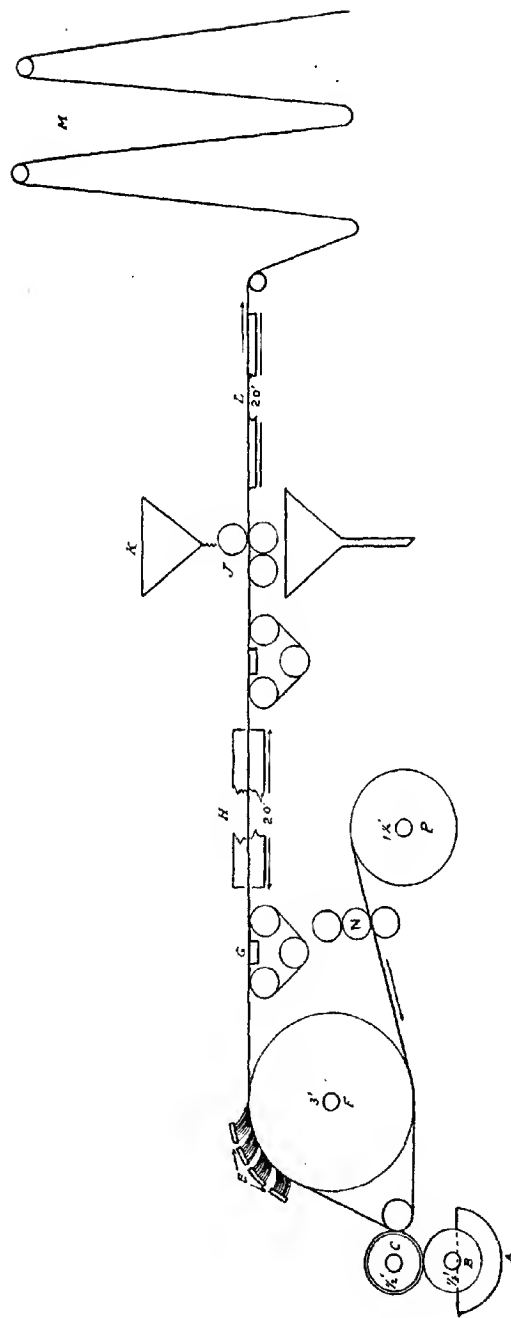
The preparation of adhesives consisting of lac or hard lac resin and unvulcanised, vulcanised or reclaimed natural or synthetic rubber has been the subject of a British Patent. Lac makes up about 40 per cent of the lac-rubber mixture, and hardening agents like urea, thiourea and hexamethylenetetramine may be used. Instead of mixing the solution in a solvent or aqueous medium, heat may be used to soften the mixture especially in plywood manufacture. Results of tests on specimens of plywood made with shellac-rubber adhesives show that the impact strength of the plywood is considerably improved as a result of the addition of rubber.

The possibility of using shellac modified with urea or melamine and formaldehyde as an adhesive in the preparation of laminated boards has already been indicated in the previous chapter. Articles prepared with this adhesive are found to have better resistance to the effect of heat and water than those in which pure shellac forms the bonding medium.

The modified shellac adhesive is prepared by heating 100 parts of shellac with 50 parts of formalin for an hour on an oil-bath at about 120°C. under a reflux condenser. 200 parts of rectified spirit (95 per cent alcohol) are then added to dissolve the mass and the resulting solution is refluxed with 15 parts of urea for another hour. The varnish is then treated with 10 parts of Santiciser 8 (*o*- and *p*-toluene ethyl sulphonamides) to improve the elasticity of the film, after which the adhesive is ready for use.

Pieces of jute or drill cloth are coated with the cold varnish either by brushing or by dipping until a sufficiently thick and uniform coat is obtained. The dried pieces of the fabric are cut to the approximate size required and placed one over the other in sufficient layers to obtain the desired thickness. The different layers are then pressed together in the hydraulic press for ten minutes at 130°-140°C. under a pressure of 1-1½ tons per sq. in. and the resulting board is removed from the press at 110°C. and cut to the exact size.

Laminated boards prepared by this method at the Indian Lac Research Institute have shown good strength and can be cut, sawed or drilled as desired. The jute fabric



L. STEEL TABLE (STEAM HEATED)

M. FESTOONS.

N. PRINTING ROLLERS.

P. PAPER OR CLOTH REEL.

F. BIG ROLLER.

G. THE VACUUM TABLE

H. PRELIMINARY BAKING CHAMBER.

(HOT AIR)

J. ABRASIVE SPREADING APPLIANCE.

K. HOPPER FOR ABRASIVE MATERIAL

A. VARNISH PAN (STEAM HEATED)

B. COPPER ROLLER.

C. RUBBER ROLLER.

D. TENSION ROLLER.

E. LEVELLING BRUSHES.

Fig. 16

COATING MACHINE FOR ABRASIVE CLOTH AND PAPER

usually gives a rougher surface compared with the cotton cloth owing to the thicker and coarser fibres and can be put to uses not requiring great beauty; articles from cloth give a much smoother surface. In either case, especially with the costlier cloth-laminated boards the appearance and the resistance to atmospheric conditions can be considerably improved by giving a covering coat with a nitrocellulose or shellac-nitrocellulose lacquer and polishing in the usual way. Smooth, glossy boards of any desired colour are thus obtained which may be used for making partitions, table-tops, etc.

Water-proof abrasive papers or cloth

The use of shellac as an adhesive in the preparation of sand paper or similar abrasive papers or cloth is rather limited on account of the fact that starchy materials, vegetable and animal proteins, etc., are very much cheaper than shellac and also because a dry coating of such substances can be rendered adhesive by merely wetting with water. But the abrasive papers made with these adhesives are not waterproof as the abrasives would come off the base when rubbed in presence of water thus limiting their use only for dry surfaces. Water-proof abrasives, on the other hand, may be used in contact with water and find wide use in metal industries, railway workshops, etc. A water-proof adhesive from shellac has been prepared at the Institute which has been pronounced to be excellent for the purpose. The composition on a commercial scale may be made as follows:

Dewaxed garnet lac (100 lbs.) is gradually added to linseed oil or castor oil fatty acids (24 lbs.) in a varnish kettle heated to 130°–140°C. and the mixture allowed to react at this temperature for 1–2 hours. The resulting product is allowed to cool to about 80°C. and dissolved in alcohol (20 gals.). To the solution so obtained is added formalin (3 gals.) and urea (6 lbs.) and the mixture boiled under reflux for 2 hours. 2 lbs. of maleic or phthalic anhydride is now added, and boiling continued for another hour after which the mixture is cooled and strained. The varnish may be brushed by hand or used in a large-scale commercial coating machine.

For the production of the abrasive paper, the paper is first rendered non-soaking by means of a light coat of heavy mineral oil and then fed into the machine in the form of a roll (Fig. 16). The varnish is placed in a pan from where it is taken up by a half dipping copper roller. The copper roller applies the varnish to a neighbouring rubber jacketed roller which applies it to the paper surface. The varnish is then levelled up by moving brushes and the coated paper then passes over a vacuum table (where it is stretched) into the preliminary drying chamber. Here it comes against a blast of hot air (from a steam box at the bottom) in the opposite direction and gets partially dried. From there, it proceeds over another vacuum table on to a set of rollers where the abrasive material is spread very uniformly on the coated surface. It then passes over a steam heated steel table when the abrasive sinks into the resin and gets set. The paper is then dried hanging in the form of festoons. After standing

overnight, another light coat of the same varnish is given over the coated surface to 'fix' the abrasive in position, and the paper is dried, cut into the desired size and baked at 100°C. for $\frac{1}{2}$ to 1 hour.

Machinery Required

	Rs.
(i) A varnish kettle, gas, steam or electrically heated, provided with mechanical stirring device through a 'gland' and a detachable reflux condensing system	2,500
(ii) Coating machine (1 side only) with printing rollers, preliminary drying box, abrasive dropping hopper and spreader, steam heated steel-table and festooning arrangement	15,000
(iii) Baking chamber, shelf-type	1,500
(iv) Reeling and cutting equipment	1,000
(v) Packing and other miscellaneous accessories, repair shop, etc. ...	5,000

As there is only a comparatively limited demand for this product, it is best that the manufacture is taken up only by such firms as are already engaged in the production of the ordinary type of abrasive papers and the like. The additional equipment required for the production of water-proof abrasives in the case of such factories will be only

- (i) the varnish kettle as described above and
- (ii) the baking chamber.

Cost Sheet

Capital investment

Machinery	25,000
Building and fittings	15,000
				Total Rs.	40,000

Cost of varnish (50 gals.)

				Rs.	A.	P.
Garnet lac (dewaxed)	160 lbs.	40	0	0
Linseed oil fatty acids	38.4 "	13	11	0
Alcohol	32 gals.	32	0	0
Formalin	4.8 "	18	0	0
Urea	9.6 lbs.	4	13	0
Phthalic anhydride	3.2 "	1	10	0
Steam and Electricity	4	0	0
Labour	2	8	0
				Total Rs.	116	10 0
or cost per gallon					2	5 4

<i>Cost of producing 10,000 sheets (9" × 11") per day</i>				Rs.	A.	P.
Paper required including 'oiling'	50	0	0
Varnish	50 gals.	116	10	0
Abrasive	400 lbs.	50	0	0
Labour	10	0	0
Steam and Electricity	15	0	0
Depreciation, Interest, etc. 15%	20	0	0
Total Rs.				261	10	0

or cost of 100 sheets: Rs. 2-9-10.

The details of the process are the subject matter of Indian Patent No. 29195.

Plywood

The use of plywood in industry, especially in packing, has been in existence only for the past 40 years and within this period the industry has grown to extremely large dimensions. Laminated veneers of wood can be obtained in large sizes from timber of even small cross-sections and built up into large sheets of such size that cannot be obtained from the same timber by ordinary sawing. Further, veneers of artistic grained woods can be used to cover thick sections of cheaper woods, as for example, panelling with oak veneers. During the last few years the fabrication of plywood has received an added impetus by its use in the manufacture of aeroplane parts and in boat building.

The principal kinds of glue used for binding or cementing veneers are :—

- (i) vegetable glues,
- (ii) animal glues such as bone and fish glues,
- (iii) blood albumen glues,
- (iv) synthetic resin glues such as phenol-formaldehyde and urea-formaldehyde resins.

For tea chests and general utility plywood, the most commonly used glue is casein glue in which the principal ingredients are casein, hydrated lime and water. Where very high resistance to moisture and weather are required, for example, in aircraft and marine plywoods, synthetic resin glues from phenol-formaldehyde and urea-formaldehyde resins are used. But synthetic resin bonded plywoods, apart from being costly are not easily available in the country owing to restrictions on imports. Besides, the raw materials required for preparing these glues are also not readily available. Attempts are therefore made to prepare a water-resistant adhesive from shellac for bonding plywood with a view to increase its utility in many industrial applications.

Two compositions have been developed at the Institute for use as plywood adhesives.

1. Lac Garnet	100 gms.
Formalin (40 %)	25 c.c.
Urea	6 gms.
Denatured alcohol	200 c.c.
Aluminium chloride	2 gms.
Fine saw dust (100-mesh)	3 "
Kaolin	20 "

Final volume made up to 400 c.c. with denatured alcohol.

Lac is at first dissolved in alcohol and to the solution are then added formalin, urea and aluminium chloride. The whole solution is then refluxed over a water bath for 2-3 hours, cooled and mixed while stirring with saw dust and kaolin. The stirred solution is then applied on the veneer which has been previously dried to contain 8-10 per cent moisture. The core is coated on both sides with the adhesive and the face veneers on one side only. The coated veneers after air-drying and baking at 80°-90°C. for one hour are arranged alternately at right angles to the grain with the core in the middle and pressed at 250-300 lbs. per sq. inch at 130°-140°C. for 8-10 minutes and then taken out after cooling down to 80°-90°C. The boards are then trimmed and finished.

Samples of plywood with the above adhesive using rotary cut veneers of *Cedrella tona*, *Mangifera indica* and *Polyalthia fragrans* were prepared and tested by the Forest Research Institute, Dehra Dun, and were pronounced to be 'quite good' as regards glue adhesion both in the dry and wet conditions. The boards, however, lack in elasticity required for air-craft plywood and further experiments are in progress to improve the material in this respect. The table of properties given below would indicate a wide range of application in many fields other than that of air-craft plywood. (See Table on page 49.)

2. (a) 10 gms. glue and 20 grams shellac are dissolved in hot dilute ammonia. 70 gms. of rubber in the form of latex are then added to the above solution and boiled for a few minutes.

(b) 5 gms. of zinc oxide, 5 gms. magnesium oxide, 10 gms. sulphur, 10 gms. urea and 0.5 c.c. tri-ethanolamine are ground in a ball mill in water to form a paste.

(a) and (b) are then mixed thoroughly by stirring. Veneers of wood are then soaked in this solution for one hour, and then removed, air-dried and pressed.

It may be mentioned that the Board of Scientific and Industrial Research, New Delhi, have also developed a water-proof adhesive for making plywood from modified shellac and rubber-shellac combinations but the details of the process are not yet known.

A recent development in foreign countries is the use of plastic wood and impregnated wood for certain industrial applications. In America especially, such wood

Timber used	Condition of pressing.	Moisture.	Glue adhesion				Adhesive of veneers according to SV3 and D.T.D. 427.	Resistance of water according to SV3 and D.T.D. 427.	Bending properties according to SV3 and D.T.D. 427.	Tensile strength lbs./sq. inch.		
			Strength lb./sq. in.	Glue failure.	Strength lb./sq. in. boiling	Glue failure				Parallel direction.	Perpendicular direction.	Parallel perpendicular direction.
1. Mangifera indica	Pressed for 8 minutes under a pressure of 300 lbs./sq. inch at 140°C. and cooled down to 90°C. before removal from press.	6.8	350	20	137	100	Passes	Passes	Passes	6525	3169	9694
2. Cedrella toona...		7.7	376	40	215	95	Do.	Do.	Fails	5391	1743	7134
3. Polyalthia fragrans		7.4	296	5	240	80	Do.	Do.	Do.	10396	5815	16211
Grade 1 commercial Plywood		...	150	...	76	7000	4000	12000
Grade II commercial Plywood according to specification No. 1. M. 2106	100	...	50	5000	3000	9000

termed 'compregnated wood' and in England 'Hydulignum' is very widely used nowadays. Shellac bids fair to be a very useful resin for the treatment of wood through surface coating, impregnation and bonding.

The impregnation of Indian woods by shellac has also been studied at the laboratories of the Board of Scientific and Industrial Research and the process has been patented in Indian Patent No. 27714.

Cost Sheet for Process 1

						Rs.
<i>Building and equipment</i>						
Building	2,000
Boiler	2,000
Peeling lathe	2,000
Veneer trimmer	1,000
Varnish still with reflux condenser (70 gals.)	2,000
Varnish spreader	1,000
Baking chamber	1,500
Hydraulic Press 10 daylight, 200 tons, 30" x 40"	3,500
						15,000

<i>Cost of preparing 60 gallons of adhesive</i>						Rs. A. P.
Garnet lac	...	150 lbs.	37 8 0
Formalin	...	37.5 lbs.	14 1 0
Urea	...	9 lbs.	4 8 0
Aluminium chloride	...	3 lbs.	3 3 0
Saw-dust	...	45 lbs.	2 13 0
Kaolin	...	30 lbs.	0 15 0
Methylated spirit	...	40 gals.	40 0 0
Labour	2 8 0
Steam and Electricity	3 0 0
Total						108 8 0

Cost per gallon ... Rs. 1 13 0

Cost of manufacturing 160 sheets of 3 ply (2' x 3') in 8 hrs.

						Rs. A. P.
Wood, 60 c. ft.	60 0 0
Adhesive, 60 gals.	108 8 0
Labour	9 0 0
Steam and Electricity	7 0 0
Depreciation, Interest, etc. 15%	7 8 0
Total cost for 960 sq. ft.						192 0 0
or cost per sq. ft.						Rs. 0 3 2

Laminated Boards

In the electrical industry, insulation boards built from layers of paper or cloth with shellac as adhesive have varied applications. The improvements desired in such boards are greater heat, water and humidity resistance. To a certain extent, synthetic resins have replaced shellac on account of their superiority in these respects, but in the special field of high voltage equipment where a high degree of resistance to surface carbonisation is required shellac is still found to be more suitable. Although laminated materials were first introduced in the electrical industry, modern requirements of various types of constructional materials have led to the manufacture of laminated boards for table tops, boxes, partitions, decorative panellings and sheets with special acoustic properties for studios, theatres, etc. A very extensive use of jute and shellac in the laminated form for motor car bodies could be envisaged in the near future. The formulation of different types of modified lac possessing improved heat and water resistance, adhesion, etc., has shown the way for their utilisation in this field. Some of the compositions described under chapter VI on shellac plastics have been utilised with different types of fillers for the production of laminated articles (cf. Indian Patent Nos. 28277 and 28281 of the Board of Scientific and Industrial Research). Below is described the detailed procedure for the manufacture of laminated boards of paper, jute and cloth, as during the last war emergency, there was an acute shortage of metals for containers.

Jute Boards

Laminated jute boards of a very high degree of finish and good mechanical properties are prepared by first impregnating jute waste with a shellac-casein solution and sandwiching the same between layers of hessian cloth also impregnated with a shellac varnish. A modified shellac varnish is then used as a finishing and protective coating over the laminated boards.

Waste jute from mills (fibre length 1 to 3 inches) is first crushed in a Christy-Norris Disintegrator down to 3 to 5 mesh and is then dried and impregnated with the resin solution as described below.

(a) Lac (*Kiri* lac) 270 lbs. is soaked overnight in a steam jacketed pan with water (67.5 gals.) containing liquor ammonia 0.88 (30 lbs.) and then warmed at 60°C. with stirring till all the lac is dissolved.

(b) Casein (34 lbs.) and water (22 gals.) are allowed to soak overnight in another steam jacketed pan (30 gals.) and then warmed to 60° C. with stirring after the addition of 4 lbs. of liquor ammonia (0.88) till all the casein is dissolved.

(c) Lime (3 lbs.) is dispersed in water (3½ gals.)

The lime suspension is at first added to the casein solution (b) with good stirring and the casein lime solution is added to the lac solution (a). At the same time, aluminium stearate (7 lbs.) finely ground is stirring into the whole solution. The solution is then further thinned with water if necessary and then mixed with aliquot parts of dry jute-waste (300 lbs.) (previously ground to 3 to 5 mesh) in a kneader mixer

and air-dried. The soaked jute is then further dried for one hour at 80°-90° C. disintegrated again to 3-5 mesh and stored.

Closely woven jute canvas (110 yds. of 40" width) is impregnated with the same varnish till the proportion of the binder to the fabric is approximately 1:1. The impregnated fabric is also dried at 80°-90° C. for one hour before use.

The impregnated jute waste is heaped over a sheet of hessian of the size of the required board and covered over with another of the same size and pressed between hot plates under a pressure of 1,000 lbs. per sq. inch at 140°C. for 10-15 minutes and taken out at 40°-50°C. after cooling. The plates' surface in contact with the materials should be highly polished and preferably lubricated at the moment of pressing, with stearic acid, paraffin or cocoanut oil. The boards thus obtained are then given a coating of finishing varnish by spraying and baked at 80°-99°C. for about one hour to make them waterproof.

The finishing varnish is made by refluxing 6 lbs. of lac with 2 gals. of denatured alcohol, 3 oz. of urea and 5 oz. of linseed oil fatty acids for 2-3 hours in a steam jacketed still.

The use of different varnishes for impregnating the cores, the sides and exterior finishing is necessitated on the ground of economy, as, otherwise, with an excellent shellac spirit varnish used all through, the cost of the finished product would be too high to withstand competition from other similar materials. The quality of laminated boards has been found to be satisfactory for many applications as they withstand 90°C. and are highly water-resistant. Samples exposed to outdoor weather condition for 6 months show practically no deterioration.

The manufacture of such boards proved profitable during the war. To keep the cost of production low, the lac used in the different formulas should be that recovered from *Kiri*.

<i>Building and equipment</i>	<i>Cost Sheet</i>	<i>Rs.</i>
Building	...	2,000
Boiler	...	2,000
C. & N. disintegrator (17")	...	1,000
Hydraulic press (10 day light, 3,000 tons, 30" x 20")	...	3,000
Steam jacketed pan (100 gals.)	...	1,500
Do. (30 gals.)	...	1,000
Do. still & condenser (5 gals.)	...	500
Mixer (universal) 20 gals. capacity	...	2,000
Drying chamber (20' x 6' x 6')	...	1,500
Spraying equipment	...	500
		<hr/>
		Rs. 15,000

Cost of preparing 160 boards (2' x 1.5' x 3/16") in 8 hrs.

(a) <i>Materials for core and outer layers</i>				Rs.	A.	P.
Lac (<i>Kiri lac</i>)	270 lbs.	42	8	0
Casein	34 lbs.	8	8	0
Liq. Ammonia (Sp. gr. 0.88)	34 lbs.	12	12	0
Aluminium stearate	7 lbs.	7	0	0
Lime	3 lbs.	0	1	6
Jute waste	300 lbs.	18	12	0
Jute canvas (40" wide)	110 yds.	27	8	0
				Rs. 116 12 6		
(b) <i>Varnish for surface-finish</i>				Rs.	A.	P.
Lac (T. N. shellac)	6 lbs.	2	4	0
Urea	3 oz.	0	1	6
Linseed oil fatty acids	5 oz.	0	2	0
Methylated spirit	2 gals.	2	0	0
				Rs. 4 7 6		
(c) <i>Total cost of production</i>						
Materials for board & finishing (a + b)	121	4	0
Labour	11	0	0
Steam and Electricity	4	4	0
Depreciation, Interest, etc. 15 %	7	8	0
				Total for 480 sq. ft.— Rs. 144 0 0		
or cost per sq. ft.				Rs. 0 4 10		

Paper boards

Fibre boards have come into increasing use during the last few years for the manufacture of suitcases, panelling, sliver cans and as a general stiffening material in various fields of application. With the availability of cheap paper it should be possible to bond several layers of them to obtain boards of satisfactory strength. An adhesive based on shellac and casein has been found suitable for bonding sheets of paper to give boards of any desired thickness.

Process

Processes for making the bonding solution and the finishing varnish are substantially the same as described in the manufacture of jute boards excepting that T.N. shellac is used in place of *Kiri lac*. For the production of laminated boards, the paper in the form of rolls is coated on both sides in a coating machine of any standard design and then passed through a steam heated drying tunnel at the rate of 1 ft. per minute and then rolled on the other end. The coated paper on the rolls is then cut

into any desired lengths and several layers of these sheets, depending upon the thickness of the board required, are arranged one over the other and, after further drying these in an oven at 80°-90°C. for half an hour, pressed between steam heated platens in the hydraulic press at 110°-115°C. for 10-15 minutes under a pressure of 1,000 lbs. per sq. inch. The platens are then cooled down to 40°-50°C. by passing cold water and the boards taken out. The boards are then finished with a waterproofing varnish, baked for $\frac{1}{2}$ hr. at 80°-90°C. trimmed and packed.

<i>Building and equipment</i>						Rs.
Building	2,000
Boiler	2,000
Coating machine and drying tunnel	3,000
Baking oven	1,000
Hydraulic Press (10 day light, 600 tons, 30" × 40")	5,000
Trimming machine	500
Spraying equipment	500
Steam-jacketed pan (100 gals.)	1,500
Do. (15 gals.)	500
Do. still and condenser (10 gals.)	1,000
						<u>Rs. 17,000</u>

Cost of preparing 160 boards (2' × 3' × 1/16") in 8 hours

<i>(a) Materials for board—</i>						Rs.	A.	P.
Unsize paper in roll (24" wide and 1,280 yds. long). (weighing approximately 180 lbs.)	45	0	0
Lac	160 lbs.	60	0	0
Casein	20 "	5	0	0
Liq. Ammonia (Sp. gr. 0.88)	20 "	7	8	0
Aluminium stearate	4 "	4	0	0
Lime	1.5 "	0	0	9
						<u>121</u>	<u>8</u>	<u>9</u>

(b) Varnish for surface-finish—

Lac	12 lbs.	4	8	0
Urea	6 oz.	0	3	0
Linseed oil fatty acids	10 "	0	3	9
Methylated spirit	4 gals.	4	0	0
						<u>8</u>	<u>14</u>	<u>9</u>

(c) Total cost of production—					Rs.	A.	P.
Materials for board and finishing ($a + b$)	130	7	6
Labour	11	0	0
Steam and Electricity	4	1	0
Depreciation, Interest, etc., 15%	8	8	0
Total for 960 sq. ft.					154	0	6
or cost per sq. ft.					0	2	7

The adhesive solutions described above for the manufacture of jute- and paper-laminated boards are prepared in aqueous medium. An alternative adhesive using spirit has also been developed from shellac for the preparation of laminated boards which do not require any finishing varnish. These boards are recommended for electrical instruments, radio parts, identity discs, etc. The preparation of this adhesive is described below :

Shellac (100 lbs.) is at first dissolved in denatured alcohol (20 gals.). To this are added formalin (40 %) (25 lbs.), urea (6 lbs.), quicklime (1 lb.) and stearic acid (1 lb.), and the whole is refluxed in a steam jacketed still, fitted with a reflux condenser, for 3-4 hours. The solution is then cooled and used as in the case of paper boards described previously. An approximate costing of the boards prepared with the varnish is given below :

<i>Cost of Laminated Boards with Shellac-Formaldehyde-Urea Varnish</i>					Rs.	A.	P.
Lac T. N.	...	100 lbs.	37	8	0
Denatured Alcohol	...	20 gallons	20	0	0
Formalin	...	25 lbs.	9	6	0
Urea	...	6 "	3	0	0
CaO	...	1 lb.	0	0	6
Stearic acid	...	1 lb.	0	8	0
Total for 33 gallons					70	6	6
or cost of varnish per gallon					2	2	2

Laminated Board of 1/16" thickness and 1ft. × 1 ft.

Varnish required 2 gallons	4	4	4
Paper	...	6 lbs.	1	8	0
Cost of 30 sq. ft. of board					5	12	4
Labour	0	8	0
Steam and Electricity	0	3	0
Depreciation, Interest, etc.	0	3	0
Total cost for 30 sq. ft.					6	10	4
or cost per sq. ft.					0	3	7

Compositions of the various types of adhesives based on lac and hydrolysed lac (*vide* appendix) and their performance characteristics from the point of view of adhesion strength to metals, investigated at Metropolitan Vickers Electrical Company in London, are given in the following tables.

Compositions of shellac-based adhesives and method of mixing

No.	Adhesive Composition (parts by weight)					Type of adhesive	Method of mixing
	1	2	3	4	5		
	Orange Shellac	Hydrolysed lac	Adhesion Promoter %	Filler	Solvent or dispersion medium		
1*	100	Cement	Melt mixed in agitated pot, roller or Banbury mixer, the filler being added after homogeneous mixing of the resinous components. (Temp. of mixing 110°-120°C., period 30-40 mins.)
2	75	25	...	Mica †	...		
3	50	25	...	Mica †	25		
4	50	20	...	Slate dust	20		
5	50	25	Malic acid	Carbon black	8	Adhesive paste	Mixed cold or just warm (40°-45°C.) in putty mill, item (3) being added in solution form using item (5).
6*	...	100	Tartaric acid	Mica †	24		
7	...	98	Malic acid		
8	...	98	Tartaric acid		
9	25	100	Malic acid	Glue	Solution of item (1) in item (5), item (4) being stirred into the above solution.
10	20	100	Tartaric acid	Slate dust	60		
11	20	90	Malic acid	Slate dust or mica †	60		
12	...	100	Malic acid	Asbestos powder	40		
13*	100	...	Tartaric acid	Glue	Solution of item (1) in item (5), item (4) being stirred into the above solution.
14	100	...	Tartaric acid	Mica †	...		
15	100	...	Tartaric acid	Mica †	122		
16	100	...	Malic acid		
17*	100	...	Tartaric acid	Mica †	100	Glue	Solution of item (1) in item (5), item (4) being stirred into the above solution.
18	100	...	Borax	Mica †	122		
			Sodium carbonate		
				

* These compositions are included as controls. † Wet ground mica (capable of leafing). Micronised mica† can also be used in place of mica†. († Micronised mica capable of more uniform spread). § Industrial methylated spirits. || Included for the sake of completeness (joints between Bristol boards are stronger than the material bonded).

Effect of adhesion-promoters on adhesion strength of metal joints

Ident No.	Type	Adhesion-promoter	Process of bonding	Adhesion strength (flexure)		
				Copper/Copper lb./sq. in.	Brass/Brass lb./sq. in.	Steel/Steel lb./sq. in.
6	Adhesive paste	(Control)	Light pressure at 150°C. for 3 hrs.	120	54	38
7	Adhesive paste	Malic acid	Light pressure at 150°C. for 3 hrs.	132	62	118
8	Adhesive paste	Tartaric acid	Light pressure at 150°C. for 5 hrs.	199	170	177
8	Adhesive paste	Tartaric acid	Light pressure at 200°C. for 1 hr.	299	152	183
1	Cement	(Control)	Melt application to hot surface (Contact pressure)	33	...	24
2	Cement	(Control)	Melt application to hot surface (Contact pressure)	41	...	27
3	Cement	(Control)	Melt application to hot surface (Contact pressure)	183	...	102
5	Cement	Malic acid	Melt application to hot surface (Contact pressure)	108
13	Glue	(Control)	Pressure of 1,000 lbs./sq. in. at 130°C. for 2 mins.	14	...	10
13	Glue	(Control)	Pressure of 1,000 lbs./sq. in. at 130°C. for 2 mins. (film glue)	24	...	19
13	Glue	(Control)	Clamp pressure at 180°C. for 1 hr. (film glue)	...	50	35
14	Glue	Tartaric acid	Clamp pressure at 180°C. for 1 hr. (film glue)	61	...	52
16	Glue	Malic acid and Tartaric acid.	Clamp pressure at 230°C. for 1 hr. (filler)	110	...	102

It will be seen from the above tables that, regardless of the type of adhesive, the addition of malic and/or tartaric acid contributes to increased bond strength. The effect is particularly noticeable in the case of bonding steel with adhesive pastes based on hydrolysed lac where threefold increase in bond strength results on addition of malic acid. It should be mentioned here that the minimum quantities of these promoters should be used in the formulation of the adhesives in order to minimise their adverse effect, particularly that of tartaric acid on water-resistance.

The possible application of these adhesives is listed in the following table.

Possible applications of shellac-based adhesives

(Tentative picture; letters A, B and C signify suitability in decreasing order of merit)

No.	Type of bonding job	Type of adhesive*															
		2	3	4	5	7	8	9	10	11	12	14	15	16	18		
1	Small rotor laminations, high operating temperature, grinding finish	—	—	—	B	—	—	—	—	—	—	—	A	B	—		
2	Small rotors, high insulation, high space factor	—	—	—	—	—	—	—	—	—	—	A	—	—	—		
3	Aluminum rotor disc laminations of meters, high insulation	—	—	—	—	—	—	—	—	—	—	A	—	—	—		
4	Core laminations of electrical instruments (e.g. coil short circuit tester)	—	A	—	A	—	—	—	—	—	—	B	B	B	—		
5	Laminations bonded to wedge shape and radial segment form (e.g. induction accelerators)	—	A	—	A	—	—	—	—	—	—	B	B	B	—		
6	Wood, fibre board, cork composition, press-board or similar absorbent material to sheet metal	B	A	—	A	—	—	—	—	—	—	B	—	—	—		
7	Paper or card board scale to metal hacks	—	A	—	A	—	—	—	—	—	—	A	—	—	—		
8	Paper, card board and cellulose materials (not too absorbent a surface) to each other	—	—	—	—	—	—	—	—	—	—	B†	—	—	A†		
9	Paper labels to glass bottles, tins, metal boxes, etc.	—	—	—	—	—	—	—	—	—	—	A†	—	—	—		
10	Absorbent surfaces to each other, to overcome glue starved joints (e.g. glazed porcelain, asbestos boards, cement concrete, etc., and the materials mentioned in row 6)	—	—	—	—	—	—	—	—	—	—	B	—	—	—		
11	Sealing cements for cracks and leaky joints in metals and castings	—	A	—	B	—	—	—	—	—	—	—	—	—	—		
12	Sealing and filling cracks in wood (e.g. furniture)	B	C	—	C	A	A	A	—	—	—	—	—	—	—		
13	Metal to metal (general) for high joint strength and resistance to impact	—	A	—	B	—	—	—	—	—	—	—	—	—	—		
14	Glazed porcelain, mending broken china, glassware, etc.	—	B	—	B	—	A	A	A	A	A	—	—	—	—		
15	Fixing handles to cutlery, mending toys, etc.	—	A	—	B	—	B	—	—	—	—	—	—	—	—		
16	Temporary joints capable of easy and clean separation (e.g. carbon strain gauges to machine parts under vibration study), high insulation	—	A	—	B	—	—	—	—	—	—	—	—	—	—		
17	Bonding glass, metals, etc. to smooth bakelite surface (general)	A	B	—	B	—	—	—	—	—	—	—	—	—	—		
18	Bonding glass window to bakelite case of meters, relays and instruments requiring easy technique; low temp. service	—	A	B	A	—	—	—	B	—	—	—	—	—	—		
19	Bonding glass window to bakelite case for tropical service (for extreme tropical climates recommend covering with moisture resistant coat in addition to curing)	—	A	B	B	—	—	—	—	—	—	—	—	—	—		
20	Capping cement (e.g. fixing bases to Radio tubes)	—	B	A	C	—	—	—	—	—	—	—	—	—	—		

* For comparison compare previous table.

† Where colour is objectionable, bleached or decolorised shellac should be employed in formulating the adhesive.

All the shellac-based adhesives, apart from possessing high bond strength to smooth surfaces, show high resistance to attack by transformer oil and petroleum type of hydrocarbons. Also their strength is not adversely affected by ageing as in the case of certain synthetic adhesives.

Glass and Porcelain Cement

(1) A cementing composition recently developed at the Institute is prepared by heating 100 parts of hydrolysed lac (*vide* Appendix) with 10-15 parts of maleic or phthalic anhydride at 130°-140°C. for 4-5 hours and then esterifying the same with about 200-300 parts of allyl alcohol. The allyl ester of the resin is then reacted with about 2 parts of benzoyl peroxide for about 2 hours under reflux and vacuum-distilled. The sticky dark brown resin obtained may be used as an adhesive as such or after thinning with rectified or denatured alcohol to the desired consistency, for joining broken glass parts, porcelain, etc., and then baked at 120°-130°C. for 4-5 hours. At the end of this period the bonding is found to be so good that long exposure to steam or even immersion in boiling water for 6 hours has practically no effect on the bond. In the laboratory, several broken glass apparatus and porcelain parts have been made serviceable by using this adhesive.

It has also been found that maleic anhydride and allyl alcohol can be replaced by phthalic anhydride and rectified spirit respectively, but the only drawback noted with this adhesive so far, is that both the reaction time and period of baking have to be increased to get the same effect as in the other.

It may be mentioned that the composition obtained as above is dark coloured, but for transparent and light coloured cements, hydrolysed bleached lac or aleuritic acid may be used. Among the applications of these compositions, the use for bonding glass to glass, glass to metal, metal to metal, moulded parts to each other or metals, etc., flexible micanite, etc., may be mentioned.

(2) Lac-glycol-ether cement is made by heating 70 parts of lac-glycol-ether and 30 parts of dewaxed lac at 120°C. for 2-5 hours. Hydrolysed lac-glycol-ester cement is obtained by heating to a homogeneous mix 20 parts of the ester with 80 parts of lac at 80°-90°C. The former cement is found useful for fixing bakelite type of mouldings to each other or to metals and the latter as a substitute for Dekhotinsky's cement. (See Chapter on "Ethers and Ether-esters of Lac" for details of their preparation).

Grinding wheels

In engineering industries, grinding wheels are necessary adjuncts. There are various types of grinding wheels according to the abrasive materials used or the binding substance which holds them together. Among abrasives may be mentioned corundum, carborundum, garnet, silicon carbide, silica, etc. The abrasive powders are generally bonded together by the use of silicates, rubber, resins, etc. and among the resins, phenolic synthetics have been found to impart much better bonding and heat resistance, while shellac has found special application for the manufacture of soft wheels for finishing very smooth surfaces. An increase in the heat resistance and adhesion quality of shellac would mean much wider use of lac in this field. The procedure to be adopted for

obtaining grinding wheels of high quality by using shellac and corundum or emery is as follows:—

A modified shellac varnish is prepared by reacting together lac, linseed oil fatty acids, formalin and urea in presence of alcohol and it is then mixed with the necessary quantity of a suitable grade of abrasive, moulded into shape and baked. The composition of the varnish is the same as that used for the manufacture of abrasive papers described in chapter VII.

Before adding the varnish to the abrasive, the latter is dried thoroughly at 100°–110°C. to ensure thorough impregnation. The abrasive powder is mechanically mixed with the varnish in such a manner that the weight of the actual resinous compound contained in the solution is 10–12% on the weight of the abrasive powder used. The mixture of the abrasive powder and the binder is air-dried and coarsely powdered. The resulting powder is then further dried at 90°–95°C. for 1–2 hours for complete removal of the solvent before moulding. By this means each individual grain of the abrasive carries a thin layer of the adhesive, and is therefore in an excellent condition for binding into a compact mass of the desired shape. The powder is moulded in a steam heated mould at 120°–140°C. under a pressure of 2 tons per sq. inch for 5–15 minutes depending upon the thickness of the piece. Before ejection of the piece the mould is cooled down to 40°–50°C. The moulded piece is then gradually baked at 80°–120°C. for 3–5 hours in order to develop greater hardness and water resistance. No further subsequent treatment is necessary besides trimming.

Materials and cost of preparing 200 grinding wheels of 3" diameter

Machinery required—

	Rs.
Hydraulic Press, 50-ton capacity with steam-heated and water-cooled platen	6,000
Moulds, 2 impressions	1,000
Baking oven	800
Tools and other accessories	200
	<hr/> 8,000

	Rs.	A.	P.
Varnish as in abrasive paper manufacture, 9 gals.	...	24	12 0
Emery Powder, 50 lbs.	...	12	8 0
Labourers (10)	...	7	8 0
Steam and Electricity	...	7	8 0
Depreciation, 10%	...	2	10 8
Interest, 5%	...	1	5 4
		<hr/> 56	<hr/> 4 0
Cost of 100 wheels	...	28	2 0

Grinding wheels prepared as above using 80-mesh corundum were tested at 3,000 revolutions per minute for grinding tool steel edges and was found to be quite satisfactory. Thus the fear of shellac wheels bursting or softening at high speeds is now completely eliminated by the adoption of a modified shellac and the baking treatment. There is a great future for the manufacture of grinding wheels and grinding stones of various types in India, with the present expansion of the engineering industries in which they fulfil an essential need.

The details of the process are the subject-matter of Indian Patent No. 29195 of 1943.

CHAPTER VIII

BAKING VARNISHES

A new composition using shellac for making baking varnishes or enamels has been recently developed by the Institute. This composition has been found to be suitable for use as protective and decorative coatings on metallic surfaces, particularly iron and steel products. The enamel may be made either transparent or opaque and of any desired shade depending upon the nature of the pigment used.

The details of making the enamel, and the approximate cost of manufacture are given below :—

Process 1

The process consists in cooking lac (40 lbs.) and red lead (10 lbs.) in linseed oil (64 lbs.) at about 200°–220°C. till complete dissolution. The resinous mass is then taken up in kerosene, white spirit, turpentine or any of the usual hydrocarbon solvents. If light-coloured dewaxed lac is used, the varnish in its films has a straw yellow colour and is transparent. Pigments or oil-soluble dyes may be incorporated to give any desired shade. Stoving is best carried out at 160°C. for 3 hours or 200°C. for 1 hour. The films obtained are hard and elastic, glossy and resistant to the usual solvents and weather.

Process 2

Clear Baking Oil Insulating Varnishes

100 lbs. of linseed oil is heated to a temperature of 170°C. and 3 lbs. of litharge and 1.5 lbs. of lime (CaO) are added. Temperature is then raised to 250°C. and maintained at this value for five minutes in order to complete the dissolution of the oxides in the oil. 40 lbs. of powdered dewaxed lac is then added in small lots taking care that the temperature of the mixture is maintained between 230°–240°C. during the addition. After all the lac has been added, the temperature is rapidly raised to 270°–280°C. and maintained in this region till a drop on a glass slide remains perfectly clear and homogeneous, and for about ten minutes thereafter. It is then allowed to cool to about 170°C. and dissolved in 11 gallons white spirit. It is filtered after cooling and tanked for a few days for clarifying.

The composition may also be used for the production of varnished insulating cloth, popularly known as 'empire cloth' and for this purpose ordinary shellac may be used in place of the dewaxed lac. It dries in less than two hours at 90°–95°C. and in a shorter time at higher temperatures. The product does not show any tendency to skin if properly stored.

CHAPTER IX

OIL VARNISHES

Due to the incompatibility of shellac with drying oils under the usual conditions of varnish manufacture, the use of shellac in oil varnishes was not hitherto possible. The soft lac (produced as a by-product in hard lac manufacture) has been found to dissolve readily in drying oils, slight warming being necessary to make the resin and oil mutually soluble, particularly in presence of some solvent. Such a solution is not likely to be a good varnish, as the formation of mixed esters does not take place at low temperatures, but excellent varnishes from such solutions can be made by heating and polymerising the components following the manner of varnish-making.

Another method of preparing oil varnishes from soft lac resin is described below :

The resin is first dissolved in cyclohexanol, and an equal amount of linseed or tung oil is added to the solution. The mixture is then gradually heated up to 160°-170°C. till all the unreacted cyclohexanol is removed by distillation and collected for use in the next charge. The function of the cyclohexanol is to reduce the acidity of the soft lac resin by esterification and to provide a suitable medium for combination between the oil and the resin. Finally, the varnish so prepared is heated up to 200°-210°C. for half an hour, cooled down to 170°C. and thinned with turpentine, after which about 1 to 2 per cent of cobalt linoleate on the weight of the varnish is added. This varnish, when applied on wood, paper, textiles and metals gives, on very short baking at 90°C., water-resistant and flexible films.

As a result of recent researches at the London Shellac Research Bureau and the Indian Lac Research Institute, a few processes for preparing lac-oil varnishes have been developed, which are described below¹ :

Process 1

5 lbs. of double-boiled linseed oil are heated to 100°-110°C., and then $\frac{1}{2}$ lb. glycerine and $\frac{1}{4}$ lb. lead peroxide are added to the hot oil. The heating is continued for 5 hours at 150°-160°C., after which the temperature is lowered to 120°C. Shellac in a powdered condition ($1\frac{1}{2}$ lb.) is now added in small portions of about 1 oz. at a time, after which the temperature of the mixture is slowly raised to 200°C., and maintained at that point for 5 minutes when all the shellac will have gone into solution. A clear drop on the glass plate will show the completion of the solution, when the temperature is lowered to and maintained at 140°-150°C. for a further two hours. After cooling, the varnish is thinned with the desired quantity of turpentine. A surface of glass or metal, brushed with this varnish, dries completely in 24 hours and water has no action on such a dried film.

¹ Processes 1 and 2 have been developed at the Indian Lac Research Institute, and Processes 3 (a), (b), (c) and (d) at the London Shellac Research Bureau.

Process 2

The apparatus consists of a copper or other cylindrical varnish kettle, provided with a lid and a stirring device and a long tubular chimney for the escape of the oil fumes.

10 lbs. of double-boiled linseed oil is taken in such a kettle and mixed with 2 lbs. of glycerine. The mixture is heated to 220°C in the course of 20 to 30 minutes and maintained at the same temperature for one hour, the contents of the kettle being kept well-stirred during the process. The cooled reaction mixture is washed twice with boiling water to remove the unreacted glycerine and then dried by heating to about 140°C.

10 lbs. of the dried product is then heated in an ordinary open kettle to about 170°C. and 6 lbs. dewaxed lac¹ added. The temperature is then rapidly raised to 260°C. and maintained at that value for half an hour. 5 lbs. of a good grade alkali-refined linseed oil (or any other grade of linseed oil) is now added and cooking at the same temperature allowed to continue for another half an hour. The product is allowed to cool to 170°C. and dissolved in 2 gallons of white spirit followed by an addition of 18.9 gms. cobalt naphthenate, dissolved in about 100 c.c. white spirit. After cooling and settling for clarification, the varnish is filtered and packed.

The composition-surface dries to touch in less than six hours and hard dries in less than 18 hours, has a very low acid value and the air-dried film shows no blush under cold water on twenty-four hours' immersion.

Process 3

5 lbs. of shellac, 1 lb. of glacial acetic acid, $\frac{1}{2}$ lb. of acetic anhydride, 2 $\frac{1}{2}$ lbs. of glycerine and 2.3 gms. of p-toluene sulphonic acid are refluxed with an air condenser on a sand bath till the acid value of the product comes down to about 27. The resulting esters are dissolved in 22.7 lbs. of linseed oil previously heated to 200°C. with $\frac{1}{2}$ lb. of litharge. The cooking is continued for 1 $\frac{1}{2}$ to 2 hours at 250°C. After cooling, the varnish can be thinned with turpentine and used.

The varnish described above dries on glass or metal in 6 hours and is resistant to water. Its scratch-hardness when air-dried is 100 gms. but on baking the film at 120°C. for 6 hours, the scratch hardness increases to 1100 gms. Both the varnishes, *i.e.*, described in processes 1 and 2, are suitable for waterproofing fabrics. A 24 hours' test with water showed neither wetting nor percolation through the coated fabric. The fabric remains supple and is, therefore, suitable for many purposes where both waterproofing and suppleness are demanded.

¹ It is important to use dewaxed lac in this and other oil varnishes because the wax present in shellac is not compatible with the drying oils and therefore separates out in the form of innumerable tiny specks in an otherwise clear film; it also lowers the hardness and water resistance of the films.

Process 4

(a) 5 lbs. of linseed oil fatty acids are heated to 120°C. and 5 lbs. of shellac are gradually added during a period of half an hour, with constant stirring. 1 lb. of glycerol is then put in, and 0.05 lb. p-toluene sulphonic acid is gradually added in three equal parts at intervals of 15 minutes each. Care should be taken to prevent frothing over as with the addition of the catalyst, the reaction is rather vigorous. The stirring is maintained at a fairly high speed. The acid value of the product comes down to 37 after heating for 7½ hours at 120°C. This time can be reduced to 3 hours by raising the temperature to 140°C. and the acid value can be reduced to about 25 by longer cooking; 0.5 to 2 per cent of slaked lime or magnesia can also be added to lower the acid value still further. Lime is first dispersed in a little of the product, the resultant mixture added to the mass, and the cooking continued for a further half-hour. In one case, the acid value of a product, initially 37, was brought down to 16 by treating with 0.5 per cent of lime.

The varnish is thinned at about 60°C. with white spirit or other thinners commonly employed in the varnish industry, after which it is clarified either by filtration or by centrifuging. It has been observed that films made from such a varnish, dry fairly quickly on exposure to the air, but drying can generally be accelerated by the addition of driers such as cobalt linoleate, naphthenate, etc.

(b) 5 lbs. of linseed oil fatty acids and 1 lb. of glycerol are heated together with 0.05 lb. p-toluene sulphonic acid at 140°C. with efficient stirring for exactly 15 minutes. 5 lbs. of shellac are then gradually added as described above, and cooking continued at 140°C. for 3 hours, when the acid value falls to 32. Liming or thinning is carried out as in the process described above.

(c) 5 lbs. of linseed oil fatty acid are heated to 120°C. and 5 lbs. of shellac gradually dispersed into the liquid. The temperature is then gradually raised to 180°C. and 1 lb. of glycerol added. The acid value after cooking for 5 hours at this temperature is about 32. This product cannot be thinned with white spirit alone. About 5 per cent of alcohol is necessary for thinning, but this depends on the time of cooking.

(d) 5 lbs. linseed oil monoglycerides are heated at 140°C. and 5 lbs. shellac gradually added. The temperature is then raised to 180°C. and 0.5 lb. linseed oil fatty acids added. After cooking for 45 minutes at 180°C. the acid value is 35.

In all the processes described above, efficient stirring and control of temperature are necessary. Using a lower temperature, a longer time of cooking may be necessary to produce a varnish of low acid value. On the other hand, if the temperature is allowed to rise above 150°C. in the case of processes 4 (a) and (b), or 200°C. in processes 4 (c) and (d), gelation may set in.

Shellac and the catalyst should be added gradually on account of the danger of frothing, as already stated.

For brushing consistency, the thinning ratio should be approximately 1 part of solid to 1.3 to 1.5 parts of thinners. The table below gives the drying times of varnishes

prepared as described above, compared with varnishes based on linseed oil and rosin or ester gum. It may be added that the varnishes which dry most quickly are also most resistant to water.

The products described are suitable as both air-drying and baking varnishes.

DRYING TIMES OF LAC-OIL VARNISHES

COMPOSITION OF VARNISH (Except where otherwise stated, these varnishes were prepared by cooking at 140°C.)	DRYING TIME IN HOURS		
	2.0% Drier ¹	2.5% Drier ¹	3.0% Drier ¹
Rosin-linseed oil varnishes (50 : 50) ...	24	10	8
(30 : 70) ...	24	17	8
Ester gum-linseed oil varnish (50 : 50) ...	24	17	8
(30 : 70) ...	24	17	8
Lac-linseed fatty acids-glycerol (50 : 50 : 10) ...	3½
(30 : 70 : 10) ..	6
Lac-linseed monoglyceride-linseed fatty acid (50 : 50 : 10) ...	12	8	...
Lac-linseed monoglycerides at 180°C. (50 : 50) ² ...	144	48	24
(70 : 50) ...	8
(100 : 53) ...	6
Lac-linseed oil fatty acid-glycerol at 180°C. (100 : 154 : 9.2) (After one of the formulæ in British Patent 468,542 of 1937)	48	8	...

Process 5

It has been found possible to esterify rosin completely with shellac in presence of castor oil. These esters are compatible with drying oils and soluble in hydrocarbon solvents. Shellac-rosin-castor oil varnishes may be prepared by either of the following two methods :—

(a) Rosin is dissolved in castor oil at 220°C. and shellac added. The reaction is allowed to proceed for 10 minutes at 220°C. and the temperature is then raised

¹ As a drier cobalt linoleate (5 per cent metal) was used in amounts shown as percentages by weight on the solids content of the varnishes.

² Soft film.

to 275°C. It is maintained there until within a few minutes of the previously determined time of gelation. Esters prepared in this way using different proportions of shellac, rosin and castor oil are all found to be miscible with linseed oil.

(b) Rosin is heated to 220°C. and shellac is added as quickly as it is dispersed. The temperature is maintained at 220°C. until within one minute of the previously determined time of gelation. Castor oil heated to 220°C. is then added and although the mixture appears to gel, particularly when the proportion of rosin is low, a perfectly clear solution is obtained when the temperature is raised to 270°C. The temperature is kept there until within a few minutes of the previously determined gelling time.

The time of gelation in both the methods increases with the proportion of castor oil to rosin and also with increasing proportion of rosin to shellac. In method (a) the time of gelation is much shorter if the reaction is carried out at 220°C. and apparently, the longer the time taken to raise the temperature from 220°C. to 275°C., the shorter is the time of gelation.

The solubility in alcohol progressively decreases during the reaction indicating a fall in hydroxyl value. Addition of xylol promotes compatibility of the varnishes with white spirit. An increase of castor oil content increases the drying time and all varnishes dry satisfactorily within 24 hours, some even drying in about six hours.

Varnishes, prepared by extending the oil length of some of the varnish esters to 2:1 and 3:1 by the addition of linseed oil, appeared to have a longer drying time than those without added oil.

It has also been found that the drying time of the pigmented varnishes is much less than that of the unpigmented varnishes. If the proportion of the pigment is approximately 5:1 of the non-volatile component of the varnish, the paint dries in a few minutes like a spirit paint. This gives a dead matt surface, which, however, produces an egg-shell gloss on rubbing with a dry rag. Even when extended with linseed oil, the pigmented product dries equally quickly, though the unpigmented varnish requires a longer time to dry. With high pigment content, the paints should be admirably suitable for quick-drying undercoats, and with low pigment contents, paints varying in gloss can be easily produced.

The cost sheets for the materials for three of the above processes are given below:

Process 1				Rs. A. P.		
Linseed oil	5 lbs. @ Rs. 2 per gallon	1	0	0
Glycerine	$\frac{1}{2}$ lb. @ As. 8 per lb.	0	4	0
Lead Peroxide	$\frac{1}{4}$ lb. As. 8 per lb.	0	2	0
Shellac	$1\frac{1}{2}$ lbs. As. 6 per lb.	0	9	0
				<hr/>		
Cost of 7.25 lbs. varnish Rs.				1	15	0
or 4.4 annas per lb.				<hr/>		

Process 3

			Rs.	A.	P.
Shellac 5 lbs. @ As. 6 per lb.	1	14	0
Glacial acetic acid 1 lb. @ As. 4-9 per lb. ...			0	4	9
Glycerine 2.5 lbs. @ As. 8 per lb.	1	4	0
p-toluene sulphonic acid 2.3 gms.	0	3	9
Linseed oil 22.7 lbs. @ Rs. 2 per gallon	5	0	0
Litharge 0.25 lb. @ As. 8 per lb.	0	2	0
			<hr/>		
Cost of 30 lbs. varnish			Rs.	8	12 6
or 4.6 annas per lb.			<hr/>		

Process 4

Linseed oil fatty acids 5 lbs. @ As. 6 per lb.	1	14	0
Glycerine 1 lb. @ As. 8 per lb.	0	8	0
Shellac 5 lbs. @ As. 6 per lb.	1	14	0
			<hr/>		
Cost of 11 lbs. varnish			Rs.	4	4 0
or 6.2 annas per lb.			<hr/>		

CHAPTER X

SPIRIT VARNISHES

Varnishes can be broadly classified under two heads: oil varnishes and spirit varnishes. The former contain a drying-oil, resin, thinner and drier and give films by the slow oxidation of the oil. The spirit varnishes are solutions of resins in volatile solvents which, by the quick evaporation of the solvents, leave a film of the resins on the coated surface. It is the application of shellac in the spirit varnish industry that will be dealt with in this chapter, the oil varnishes having been described in the previous one.

Shellac is an outstanding resin for the manufacture of spirit-varnishes; it has all the good properties of natural resins combined in one. Almost the only point against its being used solely or more extensively was its high price in the past, in comparison with other resins.

The commonest use of shellac in the spirit varnish industry is in the preparation of the widely known French polish, which is merely a solution of shellac in methylated spirit, applied by rubbing in several coats to give the beautiful gloss seen on furniture. While the finish is reasonably strong and durable for many purposes, the durability of the shellac finish can be increased by adding small proportions of certain substances called plasticisers. The cheapest plasticiser is the common castor oil, the addition of about 5 per cent of which (on the weight of shellac) will improve the gloss and elasticity of the finish. Other and better plasticisers are tricresyl phosphate and sextol phthalate, which in the proportion of 10 per cent on the weight of shellac considerably improve the quality of the varnish. Both these are imported synthetic chemicals costing about Rs. 3 per lb. at the pre-war rates. Taking the strength of the French polish as a 20 per cent solution, a gallon would need about $\frac{1}{4}$ lb. of the plasticiser for a 10 per cent addition and the increased cost on this account would, therefore, amount to about 12 annas per gallon. If castor oil is used, the additional cost will be negligible.

A more important point to consider is that the common French polish is not entirely suitable for articles like dining-tables, teapots, garden seats, etc., which come into long contact with hot plates or water. White patches on these articles, when water is not promptly wiped off, are common sights in the household.

The resistance of the shellac film to the action of water may be increased in more ways than one. Dewaxing of shellac, that is, elimination of the wax from shellac, and using this wax-free shellac for the varnish result in a film which is more glossy and somewhat more water-resistant than the ordinary shellac film. But complete water-resistance cannot be attained, and the film lacks adhesion, elasticity and hardness.

In a method worked out at the Indian Lac Research Institute and demonstrated with a pilot plant, partially dewaxed lac could be prepared by a cold alcoholic extraction of the by-products of shellac manufacture as well as from seedlac or shellac. The method is already fully described in Chapter II and it will be found that the cost of manufacture would amount to Rs. 6 per maund of garnet lac. Assuming that seedlac is worked for the manufacture of garnet lac, since the cost of manufacture of shellac is about Rs. 5 per maund of shellac, it will be seen that the partially dewaxed lac made by this process would be only slightly more expensive than a good grade shellac. The garnet lac obtained in this process, however, still contains about 1 per cent of wax.

It has been found that the following methods are suitable for preparing improved spirit varnishes :

1. Addition of 2-3 per cent urea or thiourea, and to improve the elasticity, plasticisers like tricresyl phosphate.
2. Incorporation of maleic acid up to a maximum of 5 per cent.
3. Condensation of shellac in the presence of solvents with formaldehyde and urea.

Varnishes obtained by all the above methods have been found to possess increased water- and heat-resistance. In addition, the films show greater resistance to abrasion, greater hardness, good adhesion and elasticity on wood, metal and glass surface. It has also been found that a slight baking of the films results in a considerable enhancement of all these properties.

Maleic acid-treated varnishes have been found to possess a special property in that if any alcohol-soluble dye is dissolved in such a varnish, the resulting lacquer has superior colour fastness on exposure to heat and light. As examples of practical application, it has been found that aluminium and tin foils coated with such coloured lacquers do not peel off or show cracks on repeated bending, twisting or folding, and electric bulbs dipped in the lacquer stand up to weathering without fading of colour or cracking of the films.

The methods of preparation of the above varnishes are briefly described below :

1. UREA-TREATED VARNISH

The preparation of this varnish is quite simple and consists in adding urea or thiourea to a 20-25 per cent solution of shellac in rectified spirit or denatured alcohol. The weight of the added chemical should be 2-3 per cent on the quantity of shellac used and should be dissolved completely by agitation. Addition of up to 5 per cent tricresyl phosphate improves the elasticity of the resulting films.

2. MALEIC ACID-TREATED VARNISH

Shellac varnishes of 20-25 per cent strength are prepared in industrial alcohol ; 0.5-5 per cent of maleic acid or maleic anhydride on the weight of shellac is added and refluxed over a water bath for 2-3 hours. The varnish is then cooled and is ready for application.

3. UREA-FORMALDEHYDE-TREATED VARNISH

Equal parts of shellac and butyl alcohol are taken in a suitable vessel provided with a reflux condenser and heated till solution is complete. 50 per cent of formalin (40 per cent formaldehyde) on the weight of shellac is added to the above solution and refluxed for one hour more at 120°C. At the end of this period, 15 per cent of urea (on the weight of shellac) is gradually added to the solution in small quantities at a time to prevent a quick reaction and the whole is further refluxed for a period of two hours. The resin solution is then vacuum-distilled to remove excess of solvent, formaldehyde and water formed during the reaction. At the end of the distillation, a thick syrupy resinous composition is obtained which is then dissolved in a mixture of 75 parts of alcohol and 25 parts of benzene or toluene. The varnish is then ready for application. If the dried film of this varnish on any surface (wood or metal) is baked by blowing hot air (at 60°-70°C.), it develops further heat- and water-resistance, and hot plates and tea-cups can be placed on it without any unfavourable effect. In order to obtain differently coloured surfaces, dyes and pigments can be readily incorporated in the varnish, whilst a small proportion of a plasticiser like Santiciser 8 (ethylester of p-toluene sulphonamide) improves the general finish and elasticity of the film.

4. CASHEW-NUT SHELL OIL—FORMALDEHYDE UREA TREATED VARNISH

By combining shellac or dewaxed lac with cashew-nut shell oil and then condensing the same with suitable proportions of formaldehyde and urea in presence of solvents, varnishes of considerable water resistance, flexibility, adhesion, resistance to alkali and solvents and good electrical insulation properties could be obtained. The improved varnish is prepared as follows :

Equal proportions of lac and cashew-nut shell oil are heated at 140°-150°C. for 2-3 hours and then dissolved in a solvent, preferably butyl alcohol, 6-8 per cent of urea and 25-30 per cent of formalin (40 per cent formaldehyde) on the weight of lac are then added and refluxed over a water bath for 4-5 hours. The resin solution is vacuum-distilled to recover the solvent, the excess of formaldehyde and the water of reaction. The thick syrupy resinous composition is dissolved in equal proportions of spirit and toluene or xylene. The varnish is then ready for application. Films obtained from such a varnish after baking at 120°-130°C. for about an hour or at 100°C. for about 3 hours are glossy, hard, tough and scratch-resistant.

Some of the film properties observed qualitatively are given below :—

Water	No effect for several days
Boiling water	No effect
Caustic soda	...	5-8%	No effect
Caustic Potash	...	16%	No effect
Ammonia 28%	No effect
Mixture of water, acetone, methyl alcohol, glycerine, ethyl alcohol, etc., up to 30%	No effect
Flexibility and adhesion on metals			Quite good

The dielectric strength of the varnish gives a value of 1500-1600 volts/mil at ordinary room temperature, 700-800 volts/mil at high humidity and about 1200-1400 volts/mil at 90°C.

It has also been observed that by initially adjusting the proportions of lac to cashew-nut shell oil before further condensing with formalin and urea, varnishes which are capable of giving films ranging from high flexibility and suppleness to hard and glossy films could be prepared. The flexible varnishes could be used for water-proofing fabrics, paper, etc., the hard ones for giving acid- and alkali-resistant coatings on metals, etc., and also for preparing plastic moulding compositions.

It may be mentioned that the varnishes described above are prepared from the original dark-coloured, raw cashew-nut shell oil which has been partially polymerised by heat treatment, and hence are rather dark-coloured, which may be a disadvantage for wider application. Light-coloured varnishes may also be prepared by using light-coloured cashew-nut shell oil obtained by vacuum-distilling the raw oil at 220°-230°C, under 15-20 mm. pressure.

Manufacturing Costs

A. Capital expenditure—			Rs.
Wood revolving cask (liquid light)	100
Steam-jacket still (30 gal. capacity)	1,100
Vacuum-distillation plant	3,000
Total			4,200

B. Cost of preparation—

(1) Urea-modified shellac varnish—			Rs.	A.	P.
Shellac 30 lbs. @ As. 6 per lb.	11	4	0
Urea 10 oz. @ As. 8 per lb.	0	5	0
Rectified spirit 10 gals. @ Re. 1 per gal.	10	0	0
Labour and overhead charges	0	4	0

Cost of 13 gals. varnish Rs. 21 13 0

Cost per gallon Re. 1-10-9.

(2) Maleic acid-treated varnish—

Shellac 30 lbs. @ As. 6 per lb.	11	4	0
Maleic acid 1.5 @ Re. 1-8-0 per lb.	2	4	0
Rectified spirit 10 gals. @ Re. 1 per gal.	10	0	0
Labour, overhead charges, depreciation, interest, etc.	1	8	0

Cost of 13 gals. varnish Rs. 25 0 0

Cost per gallon Re. 1-15-0.

(3) Urea-formaldehyde-treated varnish—			Rs.	A.	P.
Shellac 30 lbs. @ As. 6 per lb.	11	4	0
Formalin 15 lbs. @ As. 6 per lb.	5	10	0
Butyl alcohol 30 lbs. @ As. 8 per lb.	15	0	0
Urea 4.5 lbs. @ As. 8 per lb.	2	4	0
Rectified spirit 9 gals. @ Re. 1 per gal.	9	0	0
Benzene 3 gals. @ As. 12 per gal.	2	4	0
Overhead, labour, depreciation, interest, etc.	2	14	0
			48	4	0
Less cost of 24 lbs. butyl alcohol recovered	12	0	0
Cost of 15 gals. varnish Rs.			36	4	0
Cost per gallon Rs. 2-6-8.					

(4) Cashew-nut shell oil treated varnish					
Shellac 30 lbs. at As. 6 per lb.	11	4	0
Cashew-nut shell oil 30 lbs. at As. 6 per lb.	11	4	0
Formalin 7.5 lbs. at As. 6 per lb.	2	13	0
Butyl alcohol 30 lbs. at As. 8 per lb.	15	0	0
Urea 2 lbs. at As. 8 per lb.	1	0	0
Rectified spirit 6 gals. at Re. 1 per gallon	6	0	0
Toluene 6 gals. at As. 12 per gal.	4	8	0
Overhead, labour, depreciation, interest, etc.	2	14	0
			54	11	0
Less cost of 24 lbs. of butyl alcohol recovered	12	0	0
Cost of 15 gals. of varnish	42	11	0
Cost per gallon	2	13	6

There is a considerable accumulation of soft lac resin as a by-product in the manufacture of hard lac resin (*vide* Ch. IV). The utilisation of this by-product for preparing spirit varnishes has been made possible by modifying it into a hard and brittle product by the following methods:

- Condensing the soft resin in acetone solution in presence of phosphoric acid;
- Condensing with aniline and formaldehyde;
- Condensing with phenols and formaldehyde.

(a) CONDENSING WITH PHOSPHORIC ACID

The soft resin remaining dissolved in acetone after the separation of hard resin is concentrated by the removal of the solvent, and 2.8 parts of syrupy phosphoric acid, dissolved in acetone, are added for every 100 parts of soft resin. The mixture is then heated on an oil bath at 150°C. for 10–15 minutes and poured out. The cooled mass sets to a hard, brittle solid, having a softening point of 42°–48°C., and a melting point of 79°–80°C. This is remelted under boiling water and washed free from phosphoric acid. The product is soluble in all common solvents, and gives an air-drying, water-resistant varnish. It does not produce any greening effect on copper even on prolonged baking at 150°C.

A control experiment with the soft lac without the addition of phosphoric acid and after heating the lac for 2 hours at 150°C. (as against 15 minutes with phosphoric acid) yielded a hard mass which when dissolved in alcohol gave neither a water-resistant adhesive coating on glass nor a film on copper free from greening when baked. It is noteworthy, however, that the acid value in this case is only 71, whereas the product obtained by heating soft lac with phosphoric acid has an acid value of 100, from which a somewhat different behaviour would be expected.

(b) ANILINE-FORMALDEHYDE PROCESS

In the second method, soft resin is dissolved in 15–20 per cent of its weight of aniline on an oil bath at 120°C., cooled to about 60°C. and then mixed with an equal amount of paraformaldehyde. The temperature is gradually raised to 120°C., and heating continued at this temperature for about 20–25 minutes. The resulting mass on cooling is hard and brittle, and soluble in a mixture of alcohol and benzene giving an air-drying varnish.

The product obtained by modifying soft lac in this way had the following characteristics:

Acid value	65–81
Softening point	66°–70°C.
Melting point	78°–81°C.

It may be mentioned here that the properties of these modified resins are practically the same as those of average shellacs.

(c) SOFT LAC PHENOLICS

Condensation of soft lac resins with phenol or cresol and formaldehyde is carried out with ammonia as a catalyst by mixing equal parts of the different constituents and heating, first at the boiling water bath temperature till a thick consistency is obtained, and then at 130°C. for about 2–3 hours. The mass on cooling sets to a hard transparent resin, and has the brittleness of rosin. The product dissolves readily in a mixture of alcohol and benzene, cyclohexanol, and other high-boiling solvents. The acid value of the product is 60–70 and its softening and melting points are 70°–72°C. and 83°–84°C. respectively. The exact chemical nature of the reaction is not yet understood.

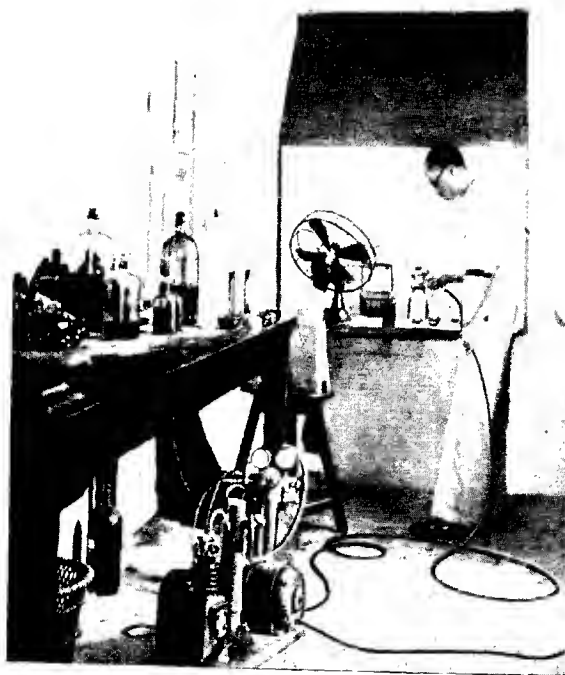


Fig. 17
SPRAY-BOOTH

The films obtained from alcohol-benzene mixture after air-drying are glossy and transparent, but not water-resistant; they also lack elasticity. Attempts are being made to improve the water-resistance and elasticity by further modification with urea. The product can, however, be readily incorporated in linseed and tung oils, when a water-resistant, flexible varnish is obtained.

5. SPRAYING VARNISHES

Shellac, or modified shellac, or mixtures of shellac with cellulose nitrate and cellulose acetate, can find application in the wider field of varnishes and clear, pigmented lacquers that are brushed or sprayed. A laboratory spray-booth (*see* fig. 17) with complete equipment, viz., a cupboard, exhaust fan, spray-gun and compressor, would cost about Rs. 750 in pre-war days.

Since spirit varnishes are applied by brushing or by the more recent method of spraying, as compared with French polish which is applied by rubbing in a number of coats, the strength of the varnish has necessarily to be more in order to give sufficient thickness of film with the application of only a few coats. In the case of straight shellac varnishes, a convenient strength for brushing or spraying is from 35-40 per cent. The addition of a small proportion of a high-boiling solvent confers better brushability or levelling property and will also serve to prevent what is technically called 'moisture blush', when the composition is sprayed. 'Moisture blush' is the whitening due to deposition of moisture on the film as a result of too quick an evaporation of the solvents, especially, in wet weather. Normal butyl alcohol is the most common and the cheapest high-boiling solvent used in this connection, an addition of 5-10 per cent of this on the volume of the varnish being found sufficient to retard the rapid evaporation of the alcohol.

As spirit varnishes find a wider application and have to withstand a greater wear than French polishes, it is also essential to use 5-10 per cent of a plasticiser in order to give the film sufficient adhesion and flexibility. As has already been mentioned, castor oil is the commonest plasticiser used, but in high-grade varnishes, it is much better to use synthetic plasticisers like tricresyl phosphate, triphenyl phosphate, sextol phthalate, etc., even though much more expensive.

The following represents a typical formula of a spirit varnish which is suitable for brushing or spraying:

Shellac	48 lbs.
95 per cent denatured alcohol or methylated spirit	9½ gals.
Butyl alcohol	½ gal.
Tricresyl phosphate	2½ lbs.

The yield will be about 140 gallons of varnish and would normally cost about Rs. 2 per gallon.

Transparent, coloured varnishes or lacquers may be formulated on similar lines but with the addition of about one-half per cent of the requisite spirit-soluble dye and

about 10 per cent of maleic acid on the final volume of the varnish ; the increase in cost amounts to about 4 annas per gallon. Such varnishes were supplied on a large scale for coating cane and wooden furniture and the reports on their performance were satisfactory.

Shellac paints or pigmented shellac varnishes can be prepared by incorporating pigments of the desired colour in varnishes of the type formulated above. These compositions are suitable for painting doors, windows and furniture and have the advantage of being very quick-drying in comparison with the usual linseed oil paints. Only two or three coats need be applied with the brush at intervals of only $\frac{1}{2}$ to 1 hour between each coat and a fine finish is obtained which will serve the purpose of both beauty and protection for the wood. For ordinary purposes, tricresyl phosphate could be replaced by castor oil or heavy pine oil, if reduction in cost is desired.

It must be mentioned here that the packing of a shellac varnish like the French polish in bulk, either for storage or for marketing, has occasionally been a vexed question. After a few months of storage in tin-plated receptacles used for the purpose, the shellac varnish is found to darken owing to the action of the acids contained in shellac on the iron exposed in places. With the best of tin-plates on the market, very small holes in the tin-coating are observed which are the sources of the trouble.

A better packing would be in tin-plate containers which have been previously coated with a thin protective film of wax ; while paraffin or any wax which is insoluble in the solvents present in the varnish would be useful to a certain extent, the best material has been found to be No. 351 of the Quaker Chemical Products Company of America.

It is also possible to avoid darkening by the addition of a small quantity of oxalic acid to the shellac varnish. 0.2-0.25 per cent of oxalic acid on the weight of lac present in the varnish would assist in storing the varnish for several months without darkening and would at the same time exert no harmful effect on the properties of the varnish.

Addition of nitrocellulose to dewaxed shellac has been found to produce a highly weather-resistant varnish. Dewaxed lac alone is capable of giving water-resistant films but the films are so poor in elasticity and adhesion that they crack and peel off on exposure to severe changes of atmospheric conditions. The addition of 10 per cent of nitrocellulose has been observed to eliminate this tendency of the film to crack.

Below are described lacquers dependent upon shellac as the major component (comprising about 83 per cent of total solids) and nitrocellulose as the minor. These have withstood weathering for more than 18 months without deterioration. Such lacquers, when applied to metal surfaces, have also the property of receiving impressions of sound grooves when pressed against a gramophone-record master in a hydraulic press. Either shellac or dewaxed lac may be used for this purpose, but considering the quality of finish aimed at, dewaxed lac, which is found to give more water-resistant films than ordinary shellac, is to be preferred. In the formulation of these lacquers, nitrocellulose

was preferred to cellulose acetate in spite of the greater sensitivity of the former to the action of sunlight, since cellulose acetate has a very limited range of solvents and the difficulty of evolving a compatible mixture of solvents is greater. Even with nitrocellulose, the alcohol-soluble variety was considered more desirable than the ester-soluble one, as the ultimate aim was to find the simplest and least expensive solvent mixture for this country, where, as yet, no proper organic solvent industry exists. Nevertheless, it has been calculated that lacquers with solvents imported in bulk and used for these preparations work out cheaper than imported proprietary lacquers. The use of alcohol in large proportions is the feature of the lacquers described below.

The formulæ of two typical shellac-nitrocellulose pigmented lacquers are given with costs of manufacture:

I. YELLOW		Rs.	A.	P.
Dewaxed lemon shellac, 80 lbs.	60	0 0
Nitrocellulose (alcohol-soluble), 16 lbs.	44	0 0
Pigment (monolite fast yellow), 5 lbs.	34	11 0*
Tricresyl phosphate, 10 lbs.	11	8 0
Butyl alcohol (normal), 1½ gals.	7	4 0
Ethyl acetate, 3 gals.	12	0 0
Ethyl alcohol (95%), 22½ gals.	22	8 0
Total		...	191	15 0

The above formula will yield 35 gallons of lacquer and the cost of the lacquer will, therefore, work out at Rs. 6-0-0 per gallon, adding 10 per cent for working costs.

II. GREEN		Rs.	A.	P.
Dewaxed lemon shellac, 55 lbs.	41	4 0
Nitrocellulose (alcohol-soluble), 11 lbs.	30	4 0
Pigments: Monolite fast yellow, 2 lbs.	13	14 0*
Monastral fast blue, 2 lbs.	24	8 0*
Barium sulphate (precipitated) 5 lbs.	0	9 0
Tricresyl phosphate, 6½ lbs.	7	8 0
Ethyl acetate, 2 gals.	8	0 0
Butyl acetate, 3 gals.	16	8 0
Ethyl alcohol (95%), 15½ gals.	15	8 0
Total		...	157	15 0

The yield will be 26 gallons and the cost of manufacture will, therefore, amount to Rs. 6-10-0 per gallon on the same basis of working costs.

* Instead of organic pigments, if inorganic pigments, say, chrome yellow and chrome green or Brinswick green are used, considerable reduction in the cost of production of the lacquer is effected. Thus, with chrome yellow in place of monolite fast yellow, the price per gallon would be reduced by about 13 annas.

It would be possible to cut down the cost considerably by employing inorganic pigments, increasing the proportion of alcohol and reducing the proportion of nitrocellulose in the above formulæ and these possibilities are now being investigated.

Comparing the above costing figures, for example, with the wholesale price of properly thinned imported lacquers at Rs. 14 per gallon, it will be found that the marketing of these shellac-nitrocellulose lacquers is an economical proposition.

The lac is dissolved in about three-fourths of the alcohol required by the formula and the pigment is mixed with the resulting varnish in a pebble ball-mill. The nitrocellulose is dissolved in a mixture composed of the esters and the remaining portion (one-fourth) of the alcohol, and the resulting solution is mixed with the shellac varnish. The plasticiser, and other solvents and diluents, if any, are then added and the finished lacquer is given another mixing in the ball-mill for a short time.

The lacquer could be sprayed or brushed, after thinning with the requisite solvent mixture to the required consistency. No gum, cotton or moisture 'blush' was met with and a fine, uniform, glossy finish could be obtained on metal surface. Successive coats could be sprayed or brushed at intervals of $\frac{1}{2}$ -1 hour.

To carry out rough tests on the durability of the finishes under actual service conditions, three coats of composition (I) were applied to the wheels of a motor car by brushing. No scrupulous attention was paid to the complete removal of the old paint and the preparation of the surface prior to painting as is done in the garages, nor was any filler or primer used. Nevertheless, a fine finish was obtained which stood up against daily use for about 20 months after which period, however, the car was not available for observation. No dullness, peeling, cracking or any other effect due to wear became apparent during the period.

Three coats of composition (II) were sprayed on an iron plate at intervals of half-an-hour between coats. As this was a rough test, no knifing glaze was used to correct the defects of the plate, nor was any special primer used as in automobile finishing. Nevertheless, a very attractive, glossy finish was obtained which was not unlike the finishes obtained with imported nitrocellulose lacquers. Compared with the film obtained from a nitrocellulose lacquer of a typical formula in a similar way, it was observed that the film of the shellac-nitrocellulose combination was slightly inferior to the nitrocellulose film in scratch-hardness, although it was sufficient for all practical purposes. Otherwise, the shellac-nitrocellulose film showed quite satisfactory properties; the film possessed good adhesion, flexibility and impact resistance, did not show blisters or any other defect on exposure to the sun every day for two months in the hot weather (maximum shade temperature being in the neighbourhood of 110°F.) and showed no 'blushing', cracking or peeling either on occasional exposure to rain or continuous immersion in water for 24 hours.

These preliminary tests were directed towards finding out whether such compositions would be suitable for first-class finishing work in actual practice and the results so far obtained have proved quite satisfactory. More extensive practical trials are now

on hand with a view to evolve the most economical finishes of this type, consistent with the desired properties for high class finishing work in general and for the preparation of sound-recording discs in particular. Recently, two motor cars have been spray-painted according to the usual garage practice with satisfactory results, shellac forming the major constituent in the primer, filler and the lacquer enamel used for the purpose.

Another modification of shellac for lacquer manufacture, which has been worked out by the New York Shellac Research Bureau, is the preparation of shellac esters and the combination of these with cellulose esters. Shellac esters are claimed to impart hardening, gloss-forming, adhesive and flexible characteristics to the lacquer and protect (like shellac) the nitrocellulose and the pigment from the action of light. These compositions have been patented and the following is a formula which represents the simplest type:

Cellulose ester (dry)	8 parts
Wetting alcohol	3 "
Methyl ester of bleached lac	20 "
Butyl acetate	24 "
Toluene	40 "

It should be mentioned in passing that owing to the necessity of processing shellac to obtain the ester and the much larger proportions of nitrocellulose and ester solvents used, this lacquer would be more expensive than the previous compositions, at least in this country.

6. PIGMENTED VARNISHES

The latest practical application of shellac which has been discovered by the London Shellac Research Bureau is in the preparation of shellac varnishes suitable for bituminous surfaces.

Two formulæ which have been found to work well are as follows:

1. Lac	25 parts
Cashew-nut shell oil	15 "
Lithopone	100 "
Industrial spirit	60 "
Cobalt linoleate	0.2 "

Cashew-nut shell oil and lac are heated together at 120°C. for one hour and dissolved in industrial spirit; this is then added gradually to lithopone and well mixed.

2. Lac (bleached)	45 parts
Industrial spirit	150 "
Sextol phthalate	20 "
Castor oil	2 "
Butyl alcohol	3 "
Lithopone	150 "

The bleached lac should contain less than 8 per cent water and the spirit less than 5 per cent water to give a satisfactory product.

It has also been reported that a certain type of hard bitumen can be dispersed in a spirit solution of lac for use as black A.R.P. (air-raid precaution) paints.

Plasticisers for Spirit Varnishes

Coatings of shellac as used in many industries are made from alcoholic or aqueous solutions. For instance, furniture polishing is carried out with shellac-spirit-varnishes, whereas leather finishes are usually made from aqueous solutions of lac.

For certain applications and uses, shellac films are rather brittle and plasticisers are generally incorporated in the shellac-spirit varnishes to increase flexibility and suppleness of the films. During an exhaustive investigation on the finding out of suitable plasticisers for lac in alcoholic solutions, it has been found that of no less than thirty plasticisers studied, only a few were efficient from the point of view of preventing crazing of the films, and imparting to them flexibility, water resistance, and resistance to hydrocarbon solvents and oils. During this study it has also been observed that the type of lac used influences the effect of any plasticiser and that, besides, the choice of the plasticiser is limited by the particular properties of the films sought for. For instance, several plasticisers are available which can prevent the crazing of the lac films and impart flexibility but are not suitable where water-resistance is imperative. Some of the plasticisers are not resistant to hydrocarbon solvents and oils so that when these properties are required in the dried lac films, such plasticisers must be avoided.

Experiments on lac solutions in industrial methylated spirit of 68 O.P. of the concentration of 20-30 per cent have shown the following to be good plasticisers:—Sextol phthalate, cyclohexanol tartrate, p-toluene sulphonamide and santiciser 1 H. In addition, softener AL, Reomal U and Reomal X may also be considered satisfactory where resistance to water is required; where only hydrocarbon resistance is required, tartrates, some citrates and plasticiser 101 (dimethyl glycol phthalate) will be satisfactory. Plasticisers like butyl stearate, butyl acetyl ricinoleate, softener AL, sextol tartrate, castor oil and diamyl tartrate do not prevent crazing.

CHAPTER XI

WATER VARNISHES

The pressing necessity to conserve the stocks of industrial alcohol in the country directed the attention of the London Shellac Research Bureau to the study of methods of dissolving lac by means of solvents other than methylated spirit, the common solvent for shellac in the manufacture of varnishes. The use of ammonia, borax, sodium carbonate, etc. is well known while the making of aqueous dispersions of lac by means of SO_2 or alkali sulphite and bisulphite is described in Chapter XII.

The use of ammonia in making the aqueous alcohol solution of lac was investigated. 100 grs. shellac are dissolved in 100 c.c. of 90-95 per cent industrial alcohol (68 O. P.) and 15-18 c.c. of ammonia solution (1 part 0.880 ammonia to 2 parts water) are added with stirring. The resulting solution is thinned with the required quantity of water, depending on the concentration and viscosity of the solution intended for application. The thinning with water in the initial stages has, however, to be done gradually and carefully. The films obtained from such solutions are clear, glossy and unaffected by water, but brittle. Films of improved flexibility, adhesion, etc. can be obtained by the addition of plasticisers (*vide* Plasticising lac films).

The above study indicates the possibility of the formulation of shellac varnishes or coating compositions with the alcohol partly or largely replaced by water. In the French polishing trade, for instance, and for other uses where application by pad on wood or other surfaces is involved, the presence of a little alcohol in the aqueous solution is desirable. This would enable adjustments to be made in the body or the viscosity of the polish, to suit individual French polishers. This technique will also reduce the tendency of 'raising the grain of the wood' and ensure sufficient anchorage for subsequent coats of polish, the final 'pull over' being done with a pure alcoholic shellac solution to get the requisite finish.

Films resistant to petrol can be obtained from the above varnish by the addition of sulphurous acid in suitable proportion. To a concentrated solution of lac in an alcohol-ammonia mixture prepared as described above and containing 100 grs. of lac is added 100 c.c. of sulphurous acid solution containing exactly 6 grs. of SO_2 . The addition is made gradually, with stirring. Some separation of the resin may occur, but this will redissolve with stirring. After standing for a few hours, there will be no odour of SO_2 . The mixture can be thinned with the careful addition of water. If separation of the resin occurs, the thinning should be postponed for some hours; if separation still occurs, the addition of a little more ammonia (of the prescribed strength) will put matters right. The sulphurous acid used should be as pure as possible. Films made from the above solution are clear, glossy and are fairly flexible when suitable plasticisers like Plasticiser 101 are added. The water resistance of such plasticised films is, however, poor, but is overcome by baking the films at 60°-80°C or by the

addition of 1 c.c. of 40 per cent formaldehyde per 100 c.c. of solution or exposure of the films to the formaldehyde vapour.

Shellac in the fur-felt hat trade

The use of shellac as a stiffening material in the wool and fur-felt industries has long been known. The solution generally used is an emulsion obtained by dissolving 100 lbs. of shellac in water containing 10-11 lbs. of borax. It has been found that 2-3 lbs. of anhydrous sodium carbonate can be used instead of 10-11 lbs. of borax for 100 lbs. of shellac in order to prepare an equally satisfactory emulsion. This not only represents an economy in quantities used, but the use of borax, which is now in short supply can thus be avoided. In the latter case, larger vessels should be employed as frothing is greater with soda than with borax. Alternatively, if it is desired to replace only a portion of borax with soda, this can be done in the following manner:—

Dissolve 5 lbs. of borax in 4-5 gallons of hot water and gradually add 100 lbs. of bleached lac. When all the lac has dissolved, mix 1.7 lbs. of anhydrous sodium carbonate dissolved in 3-4 gallons of water. Cook the brew till a clear melt is obtained and thin out with hot water to the desired consistency, which usually is 55 degrees (Barometer) at 16°C. The quantity of borax and soda may be altered at will in equivalent proportions.

It has also been found that T. N. shellac gives better penetration and gain in weight than bleached lac when the two emulsions have the same solid content. The only drawback when T. N. shellac is used is the darker colour, but for most purposes, especially where darker colours, such as brown and green, are used for dyeing the proofed wool-felt, the darker colour of the proof from T.N. emulsion is not of much consequence.

The use of good shellac (free from rosin and orpiment) possessing a maximum flow and long polymerisation-time is advisable to avoid non-stiffening of the felt during the final operation under heat and pressure. It is also advisable, whenever possible, to use softened water or rain-water for making the emulsion, as, otherwise, the presence of calcium or magnesium salts in any quantity in the water causes the formation of corresponding insoluble salts of lac which give rise to spotted or patchy appearance of the felt surface. To avoid the occurrence of skin dermatitis in hat factories, great care should be taken to avoid using shellac containing orpiment and colophony or pine rosin.

The elasticity and water-resistance of the film obtained from the aqueous dispersion described above can be considerably improved by preparing a mixed emulsion of lac/shellac and rubber.

The process of dispersing lac and rubber in aqueous alkaline media is widely known and has been the subject of numerous patents. In most cases, however, accelerators, vulcanising and dispersing agents, fillers, etc. are suggested which make the process expensive and unsuitable when cheap and ready supplies of these are not

available. As a result of preliminary work carried out at the Institute, it has been found that a cheap aqueous lac-rubber varnish can be prepared by incorporating an ammoniacal solution of rubber latex in the varnish described above followed by aerating until the rubber is thoroughly mixed. Ten per cent of rubber on the weight of lac used is found to be sufficient. Linseed or castor oil boiled with lead peroxide and pigments like iron oxide may be incorporated to give cheap and serviceable paints.

Plasticisers

Leather varnishes and dressings for leather boots and shoes have been used from time immemorial, and in spite of the introduction of more modern cellulose and synthetic resin finishes, about 60 per cent of the upper dressings in use at present are still based on shellac. The main defect found with these dressings is that the films deposited are very harsh and brittle, and peel off easily or rub off in fine powder when the shoe is handled. In order to increase the adhesion and suppleness of the lac films from these dressings, generally small quantities of glycerine, sulphonated oils, gum tragacanth, ethylene glycol, degreaser and soda oil are added to ammoniacal or borax solutions of lac, but the exact quantity and the type of plasticisers added are a closely guarded trade secret. Even films from such plasticised solutions, when they come in contact with water become brittle and lose their adhesion. A systematic study was therefore undertaken to find suitable plasticisers for aqueous solutions of lac from the dried films, of which the plasticisers would not leach out when exposed to moisture.

As the result of several experiments on the ammoniacal solutions of lac, it has been found that plasticisers like Reomol G and Plasticiser 101 (dimethyl glycol phthalate) are the best for lac aqueous solutions. The next best in order of merit are Reomol P and E. Where resistance to water is not so important, 'plastic' and diethylene glycol can also be used, as they have all the other desirable properties. Glycerine, ethylene glycol, Turkey Red oil and triethanolamine are unsuitable as plasticisers for lac.

CHAPTER XII

THE HOT-SPRAYING OF SHELLAC *.

The use of shellac bond in the production of laminated boards and tubes is well known, the shellac-coated paper being particularly suitable for very high voltage bushings. The method of coating paper with shellac hitherto has been to pass the paper through shellac varnish or to use the glue-spreader technique, the treated paper then being passed through an oven to effect evaporation of the solvent. The industrial spirit used for this purpose is expensive and inflammable, and attempts to replace this with alkaline solvents like ammonia, ethylene diamine or triethanolamine have not met with great success.

The hot-spray method avoids the use of solvents altogether as it consists in passing powdered resin at high velocity through the flame of a gun, the molten or plastic particles emerging from the flame being deposited as a film of resin on any material held in front of the flame. Factors familiar to the plastics industry, such as thermo-softening and pressure, are involved in the particles assuming a flat shape and welding together to produce a continuous film. It may, at first sight, appear that greater fire risks are involved in treating paper by this method than by the solvent process. In reality, however, with careful design of machinery, the risks are practically non-existent, the paper not even showing signs of scorching.

The experimental machine in its present form is illustrated in Fig. 18; Fig. 19 shows a sectional view of the machine and, together with the attached key, is self-explanatory. The design of a machine, such as this, involves, as in the case of all machine tools, co-ordination and control of the various feeds.

The success of depositing a uniformly thick coat of resin on paper is very much dependent upon the maintenance of an uninterrupted steady stream of powder to the gun, a condition which is particularly difficult to achieve in the case of light resinous powders containing excess of 'fines.' After trying out various feed methods, a mechanical powder-feeder was specially designed to operate in conjunction with the hot-spray machine which has ensured the necessary regularity and control in the feeding of powder. Fig. 20 and Fig. 21 illustrate the principle and constructional features of this unit. In the centre of Fig. 18 will be seen the powder feed unit mounted in position on the hot-spray machine. The powder contained in the hopper is drawn off through an adjustable gate by the revolving drum and is then transported to the barrel of the gun from the collecting funnel on the injector principle. The passage of particles through the flame is so quick (initial velocity of 100 m.p.h. at 40 p.s.i. of compressed air) that hardly any burning of the shellac particles takes place, provided particles finer than 120 mesh are excluded. Very coarse particles become insufficiently plastic to stick to paper and drop down to the collecting tray. The cost

*An article on Hot-Spray Method of coating paper with plastic material by N. N. Murty. Reprinted from 'Plastics,' June 1945.

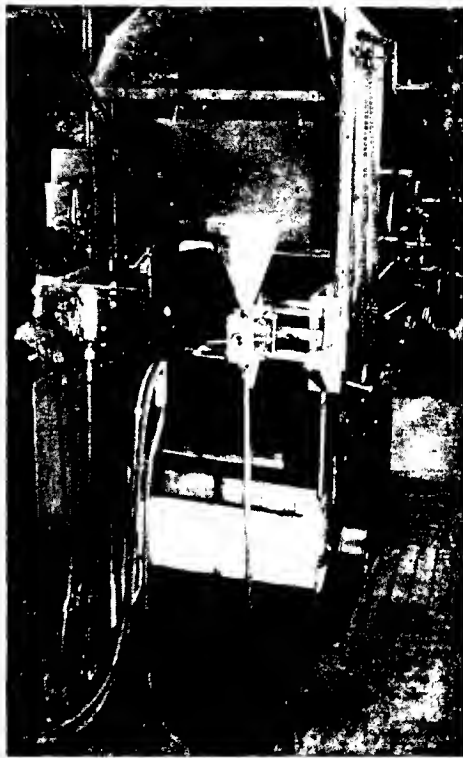


Fig. 18

EXPERIMENTAL SPRAYING MACHINE

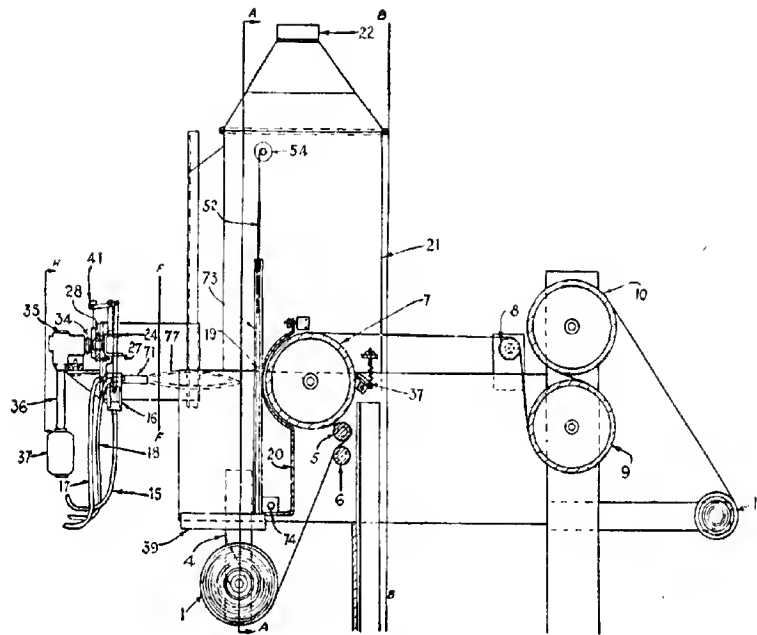
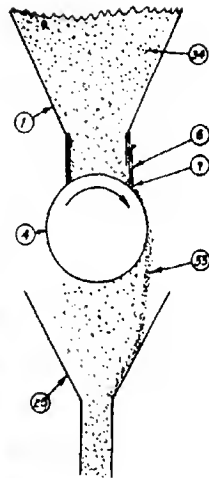
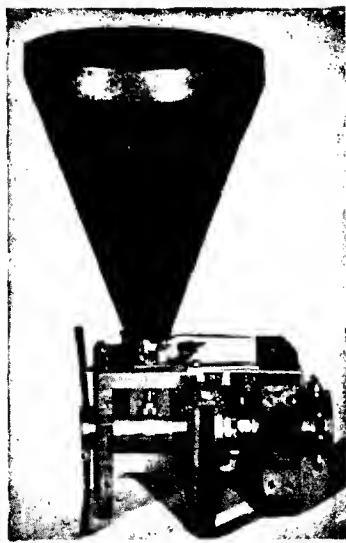


Fig. 19

SIDE SECTIONAL VIEW OF THE HOT-SPRAY MACHINE FOR COATING PAPER

1.—Roll of paper on mandrel. 5, 6, 8.—Guide roller. 7.—Spray roller (positively driven). 9, 10.—Draw rollers (positively driven). 11.—Winding roller for treated paper (friction clutch drive). 16.—Hot-spray gun. 15.—Powder feed line. 17.—Compressed air feed line. 18.—Gas feed line. 71.—Barrel of gun. 77.—Flame. 19.—Slit in guard plate (52). 52.—Drop door suspended from mandrel (54). 73.—Slotted guide bars for drop door (52). 74.—Swivel mounting for guard plate. 37.—Spring-loaded overspray scraper. 39.—Tray for receiving unsprayed and recovered shellac. 21.—Hood for extracting fumes and hot gases through duct (22). 35.—Reduction gear connected to gun feed sprocket (34). 36.—Flexible shaft driven by motor (37). 28, 29.—Guide bars for reciprocating gun carriage. 24.—Bracket projecting from gun carriage. 77.—Bracket of gun adjustably attached to lower arm of bracket (24). 41.—Plunger device operating slide door on powder feed duct in gun.



Figs. 20 and 21

FEED HOPPER AND CONTROL

1.—Hopper. 4.—Feed drum. 34.—Powder. 6.—Adjustable gate. 7.—Gate opening.
33.—Powder stream. 25.—Collecting funnel.

efficiency of the hot-spray method, therefore, is dependent in some degree on the extent to which the powder is free from coarse and fine particles. As an example, orange shellac powder, 80 per cent of which is composed of particles lying between mesh sizes 80—120, can be sprayed with less than 8 per cent overall loss under optimum spraying conditions. Microscopic examination indicates that the burnt shellac does not reach the paper but is drawn off by the flue along with hot gases. This is further corroborated in the electrical data recorded in Table 1.

Recovery and re-use of shellac deposited beyond the edges of the paper due to overshoot of the gun at either extremity of reciprocation can be effected by using side masks with a water curtain arrangement. Alternatively, the stroke or span of the gun traverse can be adjusted to lie within the width of the paper, and by means of the plunger mechanism (Figs. 18 and 19) designed to operate automatically a valve in the powder feed line, the powder supply can be shut off at the extremities of the gun traverse. Adjustment of the stroke is accomplished by altering the chain length and position of the sprockets, the flexible shaft drive permitting such adjustments.

The chain method of obtaining uniform feed motion of the gun across the paper is better suited for papers wider than 12 ins., whilst for strips of short width the cam method of feeding the gun is suitable. Better economy results, however, from treating a paper 40 ins. wide and slitting the coated paper to any desired width.

The gun used in conjunction with the machine is an adaptation of the simplified form of hot-spray gun developed by Lalkaka (Bulletin No. 5 of the London Shellac Research Bureau, 1939), which operates in a similar manner to the powder metal spray pistol, but does away with the use of oxygen and special fuels like acetylene or propane, which are not required in the case of most plastic materials.

The spray of molten or plastic shellac passing through the flame of the gun reaches the paper through a slit in the guard metal sheet. The distance between the tip of the flame and the paper is adjusted to between 2 ins.—4 ins. It is essential for the paper to be in perfect contact with the metal surface of the spray roller at the spraying point, in order to avoid scorching, burning or blistering of the paper. To overcome splitting or tearing of weak papers, the spray roller is driven positively at the same surface speed as the draw rollers. In other respects, the familiar method of feeding the paper has been employed.

Table 1.—Data Relating to the Hot-spray Method of Coating Paper with the Help of the Experimental Machine.

1. Area of the discharge end of gun barrel 12 sq. mm. ($\sim \frac{1}{10}$ -sq. in.)	4. Maximum temperature of flame when spraying 1,200°C.
2. Maximum rate of shellac sprayed through the above gun ... 3 lb./hr.	5. Maximum temperature of flame with powder supply cut off 1,450°C.
3. Velocity of powder at 1 in. from discharge end of barrel at 40 p.s.i. of compressed air ... 56 metres/sec. (~ 147 ft./sec.)	6. Length of flame at above rate of spray 8 ins.-9 ins.
	7. Distance between discharge end of barrel and sprayed material 11 ins.-14 ins.

8. Average temperature of flame along axis of flame ... 800°C.	14. Speed of coating paper at rate of 50 gm./sq. metre (1.75 mil. thick film) ... 24 sq. metres/hr. (266 sq. ft./hr.)
9. Fuel consumption (Town gas) per lb. of shellac sprayed ... ~20 cubic ft.	15. Loss through burning + unrecoverable waste ... ~10%
10. Compressed air consumed at 40 p.s.i. per lb. of shellac sprayed ... ~20 cubic ft. of free air.	16. B.D.V. (step test at 90°C.) of laminated tube 0.125 in. wall thickness wound from hot-spray shellac coated press paper... ... 60kV., (480 V./mil.)
11. Electricity consumed ($\frac{1}{4}$ h.p. and $\frac{1}{4}$ h.p. motors) per lb. of shellac sprayed ... ~0.18 unit	17. S.I.C. of above tube at 90°C. ... 4.5
12. Consistency of orange shellac powder used ... 90% lies between 80-150 mesh	18. Power factor (tan δ) of above tube at 90°C. ... 0.08
13. Speed of coating paper at rate of 86 gm./sq. metre (3 mil. thickness of film) ... 14 sq. metres/hr. (155 sq. ft./hr.)	

In order to enable cut lengths of sheet material or boards to be given a uniform coating, certain additional features have been added to the experimental hot-spray machine. The sheets or boards are fixed to the drop door (in front of the spray roller) suspended by means of ropes from an overhead mandrel, which can be wound at any required speed by connecting it to the driving gear of the spray roller (on the right-hand side in Fig. 19). In actual practice, however, it may be found desirable to limit the function of the machine to the performance of a specific task rather than adapt it for a variety of jobs.

The chief application of the machine will be in the hot-spray coating of paper intended for the production of laminated boards and tubes. Table I gives some data relating to the hot-spray coating of paper with the help of the experimental machine. It will be seen from this table that with the present size of gun a 3-mm. thick coat of shellac can be sprayed at 155 sq. ft. per hour. It is, however, sufficient for the fabrication of laminated boards and tubes to deposit about 50 grs. of shellac bond per sq. metre (1.47 oz./sq. yd.) of paper, and, furthermore, by doubling the size of the gun and employing two guns, the above speed can be stepped up seven times. This would correspond to coating 40-ins. width paper at a rate of 325 ft. per hour and, it should be stressed, this rate of treatment is achieved by a compact machine which occupies but a fraction of the space taken up by a machine having the same production rate employing solvent methods. The first cost of the machine is expected to be less than that of a machine for treating paper by conventional methods. Frequent attention to the machine can be minimized by adding cylinder capacity to the feed hopper and automatic signals and alarm devices can readily be fitted to interrupt faulty operation. Even without these automatic controls, one operator can attend to two or more machines.

Regarding running cost, it has been estimated that the hot-spray method would prove only half as expensive as the solvent method. The cost of treatment by either method is not, however, very material compared with the price of shellac.

Briefly, it may be stated that where continuous thin films free from pinholes are needed, as, for instance, in the coating of metal sheets, the solvent method is to be preferred, although with further development of plasticized shellac powders for hot-spraying there would be advantages in using the new method in this field, too. For the purpose of coating paper for fabrication of laminated products, the hot-spray method offers definite advantages. Since the hot-spray coat does not penetrate into the base material but remains on the surface, it permits pre- or post-treatment of the base material with another dielectric which may be employed for improving the electrical or thermal properties of the laminate. Furthermore, mixed resins or a mixture of fillers and resins can be sprayed which would be difficult to apply by solvent methods owing to factors such as compatibility, common solvents and settling exercising decisive limitations. Any continuous web of material other than paper, e.g., glass cloth, asbestos paper or cloth, linen or jute fabric, can be given a surface coat of plastic material or fusible bond for consolidating the treated material into laminated products. Similar advantages result in the hot-spray coating of absorbent materials such as asbestos, fuller board or wood ply with fusible bond, the possibility of glue-starved joints being eliminated.

The work described in this article was carried out by the author in the Research Laboratories of Messrs. Metropolitan-Vickers Electrical Co., Ltd., Manchester, under the scheme of co-operative research on the uses of lac in the electrical industry sponsored by the Indian Lac Cess Committee.

CHAPTER XIII

ETHERS AND ETHER-ESTERS OF LAC

1. *Lac-glycol ether*

From the present available knowledge of the nature and constitution of shellac, it is mainly a poly-ester condensation product of several polyhydroxy and polybasic acids and may be compared among synthetic resins to glyptals. Attempts were therefore made to improve or modify the properties of shellac and shellac acids by combination with polyhydric alcohols like ethylene glycol, glycol etc. and polybasic acids like phthalic, malic, adipic, succinic etc. under suitable conditions. Several modifications of these possessing improved gloss, adhesion, elasticity and water resistance were obtained. According to the nature and position of the hydroxyl and acid groups introduced, products of varying hardness ranging from a sticky balsam to a hard material could be obtained. The sticky or balsam-like products could be used as varnish, plasticisers or adhesives and the harder ones, with or without fillers, for the moulding industry.

The London Shellac Research Bureau have investigated more exhaustively the nature of the products obtained by combination of lac and lac acids (hydrolysed lac) with ethylene glycol as such or after their esterification with suitable acids like crotonic, cinnamic and oleic acids. When lac is treated with glycol, especially in the presence of suitable catalysts, an etherified product is obtained. This product which is soluble in most of the usual solvents, yields tacky and non-drying films in the cold but on baking (120°C for 3 hours) hard films which are non-tacky, highly flexible, and of excellent gloss are obtained. The lac-glycol ether so produced may also be esterified with any of the acids mentioned above.

500 parts of lac and 187 parts of ethylene glycol are heated together in a suitable vessel continuously at 120°C. until all the lac dissolves. The temperature is then allowed to fall to 80°C. and concentrated sulphuric acid (1.8 parts) added. The mixture is then heated at 180°–190°C. for 2½ hours and then poured into water. The excess of glycol and acid is then washed out repeatedly with boiling water and the product dried by heating under vacuum over the boiling water bath. The ether is readily esterified by heating with the desired acid (in the molecular proportion of 1 : 10) at 180°–200°C. for one hour in presence of 0.5 per cent of p-toluene sulphonic acid as catalyst.

Lac glycol ether can be used for a variety of purposes. It is completely soluble in the usual solvents but, when fully cured, rubber-like products are obtained which are insoluble in the same solvents and unaffected by vegetable and mineral oils. It is possible to impregnate textile and other suitable materials with these for use as packings or jointings for special purposes. The coating is non-tacky and does not require a tack-removing finishing coat. The process is simple and quicker than that of processing silk with drying oils. These treated fabrics, apart from being used as ordinary

waterproof clothing, may be used as anti-gas and perhaps as parachute fabrics. They may also be used in the formulation of paints for elastic rubber surfaces and for leather finishes. The unpolymerised compound may find further use in special adhesives.

As films from lac-glycol ether are tacky and do not harden even on long exposure to the atmosphere, stoving is essential to obtain non-tacky films. Besides, these films invariably suffer from pin-holing and exhibit sometimes other undesirable characteristics such as orange peel or bloom. Varnishes which give films free from these defects are made by partially polymerising lac-glycol ether beforehand by heating the unpolymerised compound at 120°C. for 18-24 hours and then further for 1-2 hours at 150°C. and then dissolving in a solvent mixture of 75 parts of methylated spirit (68 O. P.) 15 parts butyl acetate and 10 parts butyl alcohol. These varnishes have been found useful for many industrial applications. Briefly these are:

(i) *Electrical industry*

The excellent electrical properties of the polymerised lac-glycol ether films make them specially suitable for insulation purposes. For example, the dielectric strength of the polymerised films obtained by baking an air-dried film at about 180°C. for 2 hours was found to be 1160 volts per mil compared to 400-500 volts per mil for shellac. The highly satisfactory electrical properties coupled with great flexibility and adhesiveness of the lac-glycol ether makes it a suitable binder for flexible micanite. A sample of micanite made with the adhesive remained flexible after baking for 48 hours at 150°C. while a similar sample made with shellac-copal became brittle.

(ii) *Shellac-glyptal adhesive for insulating instrument pivots*

Ordinary shellac which is generally used for this purpose is not satisfactory due to its brittleness. It was intended to employ an oil-modified glyptal in conjunction with shellac, but these two components were immiscible in the varnish. The compatibility could, however, be obtained through the intermediary of lac-glycol ether, of which only 2 per cent is required for the resin mixtures. The new composition permitted the curing of shellac under existing baking schedule. The composition has been successfully used in the manufacture of meters.

(iii) *Coating compositions*

Adhesion of lac-glycol ether stoved films is extremely good for all surfaces e.g. paper, textiles, tin plate, steel, black iron plate, aluminium (Duralumin and Alclad) and glass. The films are elastic and resist continued compression and extension. It has been found that tin and black iron plates lacquered with lac-glycol ether varnish by the standard methods and then baked at 195°-200°C. for 22 minutes could withstand punching and pressing without any blemish. Fully polymerised films are unaffected by alternate heat and cold and resist the action of dilute acids, alcohol, hydrocarbon solvents (both aliphatic and aromatic) or a mixture of these, weak alkalis, 10 per cent solution of common salt and copper sulphate. Resistance to pool petrol is fairly good

but resistance to caustic alkalis, chlorinated hydrocarbons, ketones and ester type of solvents, and benzyl alcohol is poor.

Cans made from tin or black iron plate previously lacquered with lac-glycol ether can be thus used for packing various chemicals and solvents. Such cans may also be used for packing those paints and varnishes which do not contain either a chlorinated solvent or a mixture of solvents which cause swelling and dissolution. These cans may also be employed quite safely for packing bleached shellac spirit varnishes as no discoloration has been observed on storage for over six months, but they are, however, found unsuitable for packing acid fruits and meat.

(iv) *Resistance to lubricating oils and greases*

Stoved films of lac-glycol ether were unaffected by both mineral and vegetable lubricating oils. Resistance to calcium soap base and sodium soap base greases was good, but the effect of the greases on the films was to harden them. Resistance to cutting oils was, however, found to be not very satisfactory.

(v) *Air-drying lacquers*

Air drying lacquers can be made by the addition of either shellac or cellulose nitrate to the lac-glycol ether. In the former, the varnish is prepared by dissolving the lac and unpolymerised compound previously heated for 3 hours at 150°C. in a solvent mixture of 75 parts of methylated spirit, 15 parts of butyl acetate and 10 parts of butyl alcohol. The addition of at least of 25-30 per cent cellulose nitrate to partially polymerised lac-glycol ether is necessary to give an air drying lacquer. A typical composition is :

	<i>Parts by Weight</i>
Partially polymerised lac-glycol ether	... 20
$\frac{1}{2}$ sec. cellulose nitrate	... 6
Methylated spirit	... 48
Butyl acetate	... 18.5
Butyl alcohol	... 7.5

This lacquer has excellent adhesion on vulcanised rubber surfaces and gives very glossy films.

Lac-glycol ether has also been found to be an excellent plasticiser in shellac varnishes of the stoving type.

(vi) *Oil-silk industry*

Lac-glycol ether varnishes can be successfully employed for the production of oil-silk. The impregnated silk needs to be stoved at 120°C. for 2-3 hours when a non-tacky oil-silk is obtained which is comparable in all respects with ordinary oil-silk. The oil-silk from lac-glycol ether has of course the advantage that a tack-removing finishing coat is not necessary. This type of oil-silk has been found to withstand the

action of boiling water for half-an-hour and is unaffected when immersed for 48 hours in a solvent mixture of 80 parts by volume of methylated spirit and 20 parts toluol.

(vii) *Rubber and rubber substitutes*

When lac-glycol ether is polymerised in bulk, a rubber-like mass is obtained. This may be sheeted out and calendered on a textile base to give 'rubberised' cloth which may be used for printing blankets. Alternatively, a concentrated varnish of the partially polymerised compound may be used for the production of the same article. In this case, however, several coats would be necessary, and each coat would require baking. Fillers, pigments, and dyes may be added, and the fully coated fabric may be further calendered and embossed to produce artificial leather of 'Rexine' type.

The rubber-like polymerised lac-glycol ether may be worked on a rubber-mill with or without the addition of rubber and other ingredients, and is claimed in an American Patent to be unaffected by water or petroleum hydrocarbons. (S. Captan, U.S.P. 2,301, 253 dated 16th Nov. 1942).

2. *Substitutes for alkyd resins*

Compositions claimed to be good substitutes for alkyds in so far as they satisfy all the requirements of the alkyds, namely retention of flexibility during ageing, good electrical properties etc. can be made from lac, a dihydric aliphatic alcohol such as ethylene glycol and a monobasic acid. The acid may be either a resin acid, e.g. rosin, a simple aliphatic long-chain acid such as oleic acid or a mixture of acids, such as the fatty acids of linseed oil or cotton seed oil.

The manufacture of these products consists of two distinct stages: (a) the preparation of the lac-glycol compound, and (b) its esterification by rosin or a fatty acid.

(a) The lac-glycol compound is prepared by refluxing a homogeneous melt of lac (100 parts by weight) and ethylene glycol (38 parts) in an oil bath at 200–205°C. for one hour using concentrated sulphuric acid (0.69 parts) as catalyst. The acid value at this stage is reduced to less than 5.

(b) The lac-glycol compound thus obtained is further heated at 200°–205°C. for 30 minutes (with condenser removed) to remove any uncombined glycol. The fatty acids, e.g. linseed oil fatty acids (100 parts) are then added and the esterification is carried out in the same open vessel by heating for 4–5 hours at 150°–200°C. Thus the preparation of lac-glycol fatty acid complexes can be completed in 5–6 hours.

After esterification is complete, the esters need further heat treatment in order to reduce their 'curing' time as well as to get the optimum film properties. Films of unboiled esters have poor water-resistance and require a larger quantity of drier to become non-tacky; they have also a tendency to pin-hole on rapid baking. The heat bodying of these esters should be carried to the maximum possible degree compatible with their solubility. This is best achieved by heating with stirring at 150°C. for 18–24 hours or for 8–12 hours at 200°C. The lower temperature is to be preferred as larger molecules are thus built up and skinning is reduced to a minimum.

The thinner usually employed is white spirit. In the case of the rosin esters it is preferable to use a mixture of xylol and white spirit (1 : 2 by volume). They are compatible with drying oils, bodied or unbodied, in all proportions.

The varnishes from lac-glycol-fatty acid complexes are mainly of the stoving type, the minimum time required to give tack-free films at 150°C. and 180°C. for some of the compositions without drier varies between 60 and 40 minutes and with a drier like cobalt linoleate (0.1% of metal on non-volatile matter) 45 and 25 minutes, respectively. The film properties of the lac-glycol-linseed oil fatty acid complex (which contains 50% fatty acids) after baking at 180°C. for half an hour compares very favourably with commercial alkyd compositions as regards hardness, water-resistance, retention of flexibility after ageing at 150°C. for 200 hours and electrical properties.

When oleic acid, instead of linseed oil fatty acids, is used for esterification of the lac ethylene-glycol compound, a complex is obtained which is particularly useful where retention of flexibility at high temperatures is required. It has been established that films from this complex are superior to a long oil alkyd in regard to this property.

Air drying varnishes are obtained from lac-glycol esters of rosin or fatty acid having conjugated double bonds (fatty acids of tung or dehydrated castor oil). The linseed oil-fatty acid-ester is a general-purpose varnish. Oleic acid and cotton seed fatty acid esters are particularly recommended for electrical purposes; their excellent electrical properties, coupled with their ability to remain flexible on prolonged exposure to high temperatures make them definitely superior to many a commercial alkyd resin.

The varnishes from lac-glycol-fatty acid esters can be readily pigmented, but as all the varnishes are dark in colour, only deep shades of enamels can be made.

The processes described above are covered by British Patent Application No. 15896.

3. *Glycol ester of hydrolysed lac*

It has been mentioned that lac-glycol ether could be used as a plasticiser of lac, but the films so plasticised need to be baked before resistance to hydrocarbon solvents is obtained. Further, lac-glycol plasticised films are not sufficiently flexible at low temperatures. It has been found that when water-insoluble shellac acids are esterified with ethylene glycol a product is obtained which can be used as a plasticiser for lac to give films having flexibility at low temperatures and resistance to hydrocarbon solvents on any metallic surface, e.g. black and tinned iron, aluminium, brass, copper etc.

The glycol ester of lac acids is prepared by refluxing 500 parts lac acids with 150 parts ethylene glycol without any catalyst for 24 hours at 195°–200°C. when practically a neutral product is obtained. The ester is then washed free from glycol or any other water-soluble matter by repeated washing with boiling water and dried. It is a dark-red sticky balsam with a specific gravity of 1.08–1.095 at 20°C. It is soluble in all proportions in alcohols, esters and ketones but insoluble in ether, paraffin

and coal-tar hydrocarbons and chlorinated hydrocarbons. For use as a plasticiser for lac, about 20-25% of this ester is necessary, but for obtaining films which remain flexible at low temperatures, a much greater percentage of the ester (400 per cent) is necessary.

Lac films which remain flexible at as low a temperature as -25°C . are made as follows :—

The ethylene-glycol ester of hydrolysed lac (80 parts) is heated to 120°C . and dewaxed lac (20 parts) is slowly stirred in. When all the lac is melted, the temperature is raised to 150°C . and maintained there for 4-7 hours. The homogeneous mixture thickens considerably, but remains soluble in alcohol. The molten mass may be used as such for coating fabrics or the fabric may be either brushed, dipped or roller-coated in alcoholic solution and subsequently slightly baked at 80° - 90°C . for about half-an-hour. Detached films from the molten mass are non-tacky and can be double-folded at -25°C .

Films which are flexible and resistant to chlorinated hydrocarbons are obtained when equal parts of the ester and dewaxed lac are heated together at 150°C . for 2-3 hours and the resulting mass dissolved in methylated spirit. Unbaked films are resistant to methylene chloride at ordinary temperatures and pressures but baked films (150°C . for 1 hour) are unaffected by boiling chloroform for $\frac{1}{2}$ hour. The adhesion of these films on vulcanised rubber and neoprene surfaces is excellent and the flexibility is also very good. The ester may also be used for binding flexible mica sheets.

CHAPTER XIV

INSTANTANEOUS SOUND RECORDING DISCS

The familiar gramophone records made out of shellac and the more recently, developed records made out of cellulosic and other compounds reproduce excellent copies of the original music or speech but their manufacture involves a series of complicated and expensive processes. The recording is first taken on a wax disc of special composition, and then transferred by electroplating into a metallic negative called the master which is then used to stamp the records in a hydraulic press. It becomes evident, therefore, that these gramophone records could be marketed at a comparatively low price only by selecting the most popular music and turning them out in thousands.

There is another field of sound recording, however, in which a need has been recently created of making only a single or at the most a few records by a speedy, simple and inexpensive process. This aspect of sound recording has been made possible by the discovery of special compositions which directly take an electrical recording of sound like the wax disc but which could be played back at least a few times after recording.

These instantaneous recording discs, as they are called, are of various types. Several years ago, grooved discs had been on the market which were made of a special metallic composition, the recording being done by singing into the sound-chamber of the gramophone while the disc was revolving. As the disc, however, reproduced more the scratching noise of the needle than any music or speech, it had only a brief existence.

The real instantaneous recording blanks of quality are however made by generally coating aluminium discs with special compositions containing plasticised cellulose acetate and such discs are the 'Presto', 'M.S.S.' and others. A core of laminated paper or glass is also a feasibility and transparent, moulded discs had also been marketed under the name of 'Decility'. All these blanks can be electrically recorded with the usual equipment and give, without any processing, an excellent reproduction of the original sound. Discs of this type are used for recording one's own speech or music for amusement or educational and propaganda purposes, and by broadcasting stations for recording music or speech and re-broadcasting the same later.

Though shellac produced the best type and the largest number of gramophone records in the world, the resin had not so far found any use for instantaneous sound recording. Experiments were therefore undertaken in this line and the following

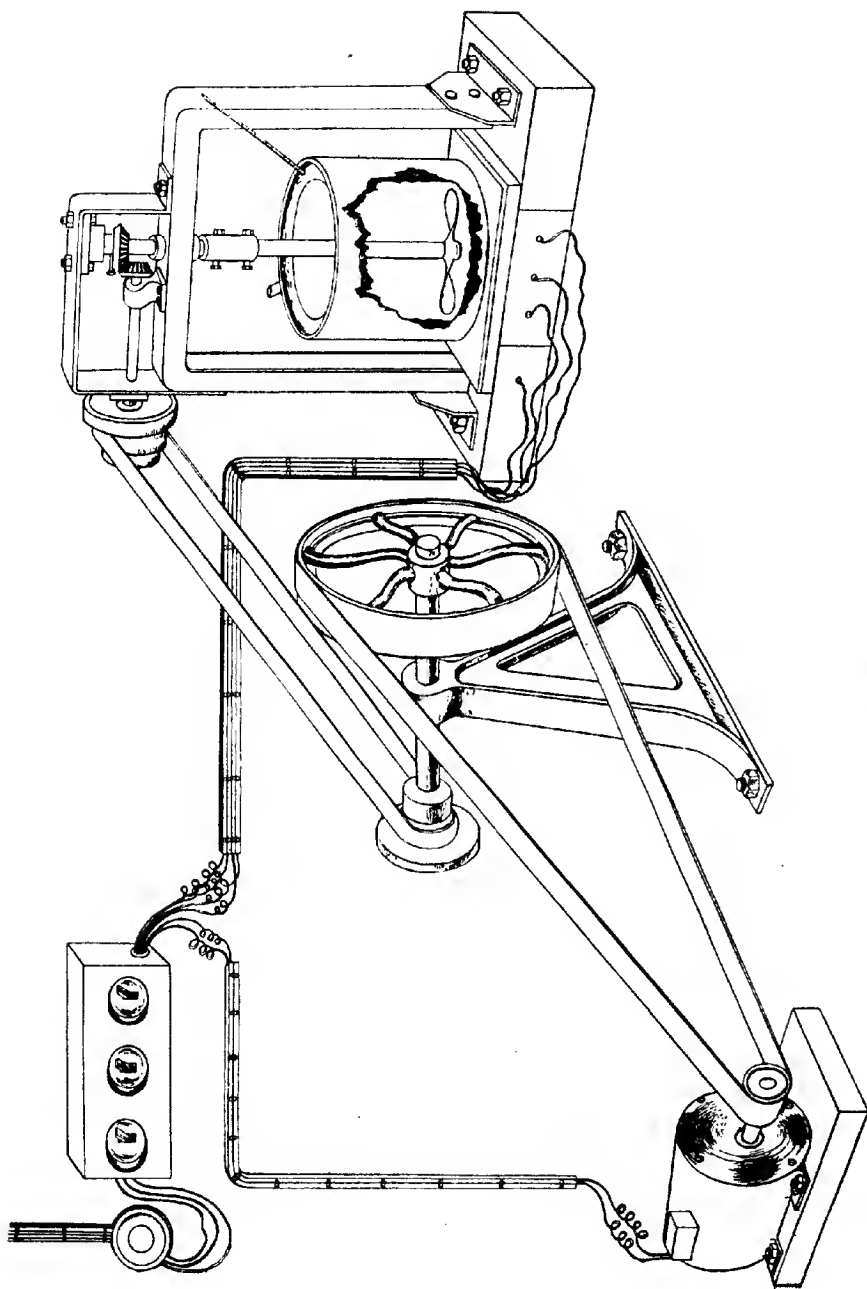


Fig. 22
VARNISH KETTLE

method describes a composition satisfactory in practically every respect except for a slightly higher surface noise.

The method consists in reacting dewaxed lac with castor oil fatty acids and glycerine and dissolving the resin thus produced in ammoniacal water. For this purpose an open varnish kettle (see fig. 22) is most convenient. 12.5 lbs. of the dewaxed lac is added in portions to 3.75 lbs. of the castor oil fatty acids heated to about 130°–135°C. in the varnish kettle and kept vigorously agitated. After all the lac has been added, the temperature is allowed to rise to 150°C. and 1.25 lbs. of glycerine added and heating continued at 155°–160°C. with stirring till the acid value of the melt falls down to 45–50. Heating is then discontinued and the temperature allowed to fall as quickly as possible to about 100°C. and the resinous mass dissolved in redistilled spirit (1 gallon). The alcoholic solution is then poured into well-agitated distilled water (3.25 gals.) containing 1 lb. of ammonia (0.888). In another vessel 12.5 lbs. of finely powdered dewaxed lac are dissolved in 3.75 gallons of distilled water containing 1.25 lbs. of ammonia (0.888). The two varnishes are now mixed and 0.25 lb. of a suitable dye is incorporated. The varnish is strained, while still hot, through several folds of the finest muslin and allowed to stand in a tall container for a couple of days at least before use.

The application to the aluminium or glass base is best done on a coating machine (see Fig. 23) inside a dust-free room, preferably air-conditioned. For this purpose the glass or aluminium disc is allowed to spin horizontally at about 20 R.P.M. and the thick varnish allowed to flow in a continuous thin stream on the surface of the disc on a travelling guide from a separating funnel from a point about $1\frac{1}{2}$ " from the centre of the disc taking care that no air bubble gets enclosed in the film during the process. As the varnish container travels more and more towards the edge, the flow of varnish is increased. When the coating is complete, the disc is allowed to dry in a perfectly horizontal position supported only at the centre. When the film has dried, the disc is inverted and the reverse side coated exactly as before. A twelve-inch disc takes about three minutes and a ten-inch disc $2\frac{1}{2}$ minutes for coating each side. After air drying, the discs are baked in a well ventilated hot air oven (Fig. 24), the discs being supported in a vertical position on a slowly revolving horizontal shaft and retained in position by separators. The baking is continued at 60°–65°C. for three hours and then at 90°–95°C. for 4 hours. A very glossy and smooth film results.

The discs may be used for recording on any of the standard recording machines. The swarf comes off smoothly and continuously and does not stick to the recorded surface. The recording is generally as faithful as on any other standard recording disc and keeps indefinitely without cold flow or distortion. The recorded discs may be hardened to prolong their 'life' by baking at 90°–95°C. for 2 hours and then may be preserved in the familiar paper envelopes and played a number of times on the gramophone or electric turntable with the usual straight needle.

Machinery required

Rs.

(i) Open varnish kettle, gas, electric or steam heated, capable of being heated to 160°C. (oil jacketed if gas or electric heating is adopted). Complete with mechanical stirring device and drain cock at the bottom (including stirring motor)	...	1,500
(ii) Disc coating machine 10 units	...	4,500
(iii) Drying Chamber complete with revolving spindle and drive mechanism	...	3,500
(iv) Miscellaneous accessories	...	1,500
	Rs.	11,000

Capital expenditure

Machinery	...	11,000
Building including air-conditioning unit	...	14,000
	Rs.	25,000

Costing data

Factory producing 500 discs per working day of 8 hrs.

Materials for 10 gallons of varnish

		RS	A	P
Dewaxed lac	... 25 lbs.	18	12	0
Castor oil fatty acids	... 3.75 lbs.	1	6	6
Glycerine	... 1.25 lbs.	0	10	0
Rectified Spirit	... 1 gallon	1	0	0
Ammonia	... 2.25 lbs.	0	13	6
Distilled water	... 7 gallons	1	12	0
Dye	... 0.25 lbs.	1	0	0

Total cost of materials for 10 gallons

Rs. 25 6 0

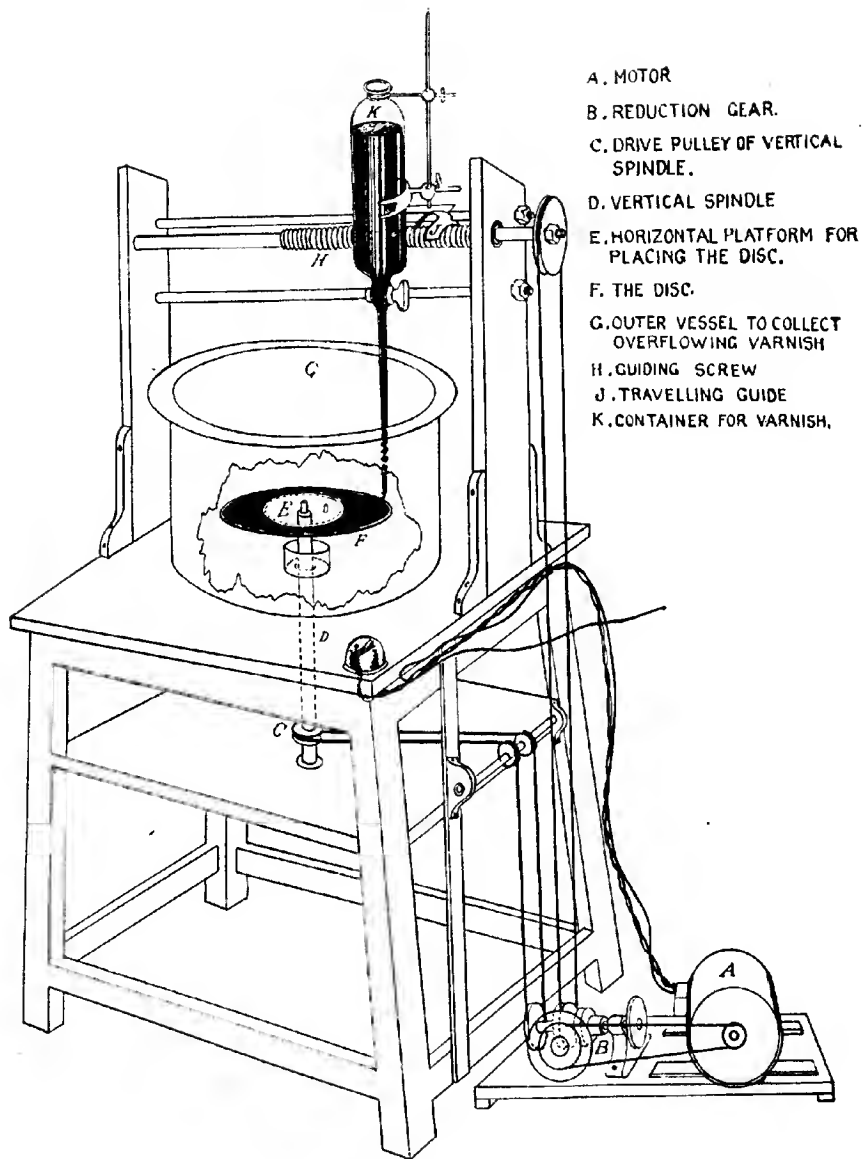
Recording Discs

		Rs	A	P
10" Aluminium blanks	... 500	125	0	0
Materials for varnish	... 10 galls.	25	6	0
Labour	...	11	4	0
Gas, steam and/or electricity	...	5	0	0
Depreciation, Interest, etc.	...	12	8	0

Total : Rs. 179 2 0

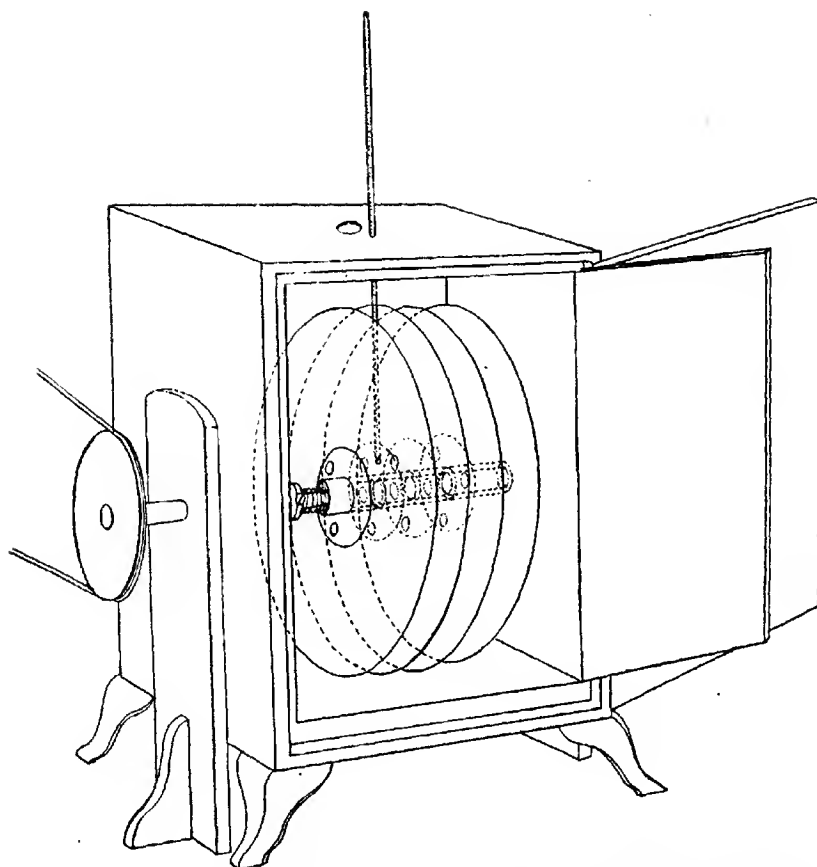
or cost of production per 10" disc ... Rs. 0 5 8

The details of the above process are the subject matter of Indian Patent No. 29193.



DISC COATING MACHINE

Fig. 23



BAKING OVEN FOR RECORDING DISCS

Fig. 24

CHAPTER XV

OIL CLOTH

Oil cloth, floor cloth and like materials find extensive use both in the home and in the trade as covering for upholstery, vehicle hoods, etc. The cheapest form of these materials is generally made by coating fabrics with linseed oil into which necessary fillers and pigments have been incorporated. For superior products, cellulosic materials, oil-modified resins and synthetic resinous materials are used. The product under description involves the use of the natural resin, shellac, incorporated in linseed oil in the presence of litharge, the last material acting both as an incorporating agent and as a drier for the oil.

The process consists in heating the linseed oil¹ (100 parts) to about 170°C. and adding litharge (6 parts) and raising the temperature to 240°C. The product is maintained at this temperature so as to enable the litharge to go completely into solution in the oil. Powdered shellac (40 parts) is now added in small lots with vigorous stirring taking care that the temperature remains between 230°-240°C. during the addition. There may be considerable frothing during the additions and this has to be beaten down before the subsequent lot of shellac is added. After all the shellac has been added the temperature is rapidly raised to 270°C. A drop of the material at this stage or after a few minutes will appear clear and bright and free from any resinous specks when dropped on a glass surface and viewed by transmitted light. The product is maintained at the same temperature for a further 15 to 20 minutes and allowed to cool to 150°-170°C. and filtered while still hot.²

The product on cooling becomes a thick semi-solid mass and after removing any 'skin', if formed, is mixed with fillers like barytes (50-80 parts) and pigments (50-20 parts) in an edge-runner so that a thick dough-like material is obtained. This forms the coating material for the fabrics.

The most suitable type of fabric must be used. This is first 'singled' or 'gassed' to burn off all protruding fibre ends and then starched and calendered to a smooth finish. The coating material prepared as described above is then applied on to the prepared surface of the fabric in any suitable coating machine provided with a doctor blade, the desired thickness of film being applied in one coat. A proper consistency of the coating material has to be maintained as too thin a material will soak through the cloth giving an unsightly appearance on the reverse side, and too thick a material will be difficult to apply and the adhesion of the dried film on to the basic fabric will be poor.

¹ Any grade of linseed oil may be used but the alkali-refined oil gives the best result.

² If allowed to cool to the room temperature, filtration will be extremely difficult owing to the high viscosity of the material as well as the precipitated lac wax finely suspended throughout the product.

After the fabric is coated, it is kept exposed to the atmosphere for three to four days or even one week depending upon the temperature and humidity of the room. For superior gloss, the coated fabric may be allowed to pass through a baking chamber maintained at 90°-95°C. for 20 to 30 minutes immediately after coating and then air-dried. The film becomes dry to touch within a few hours of coating and might take three to four days or even a week to become quite hard depending upon the film thickness and weather conditions. By suitably adjusting the proportions of cobalt drier and film thickness, very attractive wrinkle finishes may be obtained.

In excessively humid weather, it may be advisable to apply a fine dust of talc on to the coated surface before reeling it off, though this will not be ordinarily necessary.

Equipment.—

					RS	A	P
Disintegrator	1,000	0	0
Varnish Kettles	2,000	0	0
Hot filter for vehicles	1,000	0	0
Pigment sifter	2,500	0	0
Edge runner	4,000	0	0
Coating machine	20,000	0	0
Gassing and Starching machine	10,000	0	0
Reeling machine	1,000	0	0
Boiler	3,000	0	0
Workshop and Sundries	5,500	0	0
Total				...	50,000	0	0

Land and Buildings.—

Land	3,000	0	0
Factory Building	50,000	0	0
Staff quarters	40,000	0	0
Furniture and fittings	7,000	0	0
Total				...	1,00,000	0	0

Capital Expenditure.—

Machinery	50,000	0	0
Land and Buildings	1,00,000	0	0
Working Capital	1,00,000	0	0
Total				...	2,50,000	0	0

Staff Required.—

					Rs	A	P
Chemist Manager	6,000	0	0
Asst. Chemist	1,800	0	0
Chief Mechanic	1,800	0	0
Asst. Mechanic and Electrician	900	0	0
Head Clerk and Cashier	1,200	0	0
Accountant	900	0	0
Stores Clerk and Typist	900	0	0
Watchman, Durwan and Time Keeper	1,080	0	0
Labourers (10)	2,250	0	0
Other miscellaneous hands	3,170	0	0
Total				...	20,000	0	0

Recurring Expenditure.—

Salary of Staff	20,000	0	0
Publicity	3,600	0	0
Fuel and Power	5,000	0	0
Interest on investment @ 4%	10,000	0	0
Depreciation on building @ 2½%	2,500	0	0
Depreciation on equipment 10%	5,000	0	0
Contingencies	3,900	0	0
Total				...	50,000	0	0

COST OF PRODUCTION (YEAR'S OUTPUT OF 150,000 SQ. YDS. OF OIL CLOTH
52 TO 54 INCHES WIDE)

Linseed oil	750 mds. @ Rs. 16 per md.	12,000	0	0	
Shellac	300 mds. @ Rs. 30 per md.	9,000	0	0	
Litharge	45 mds. @ Rs. 20 per md.	900	0	0	
Barytes	800 mds. @ Rs. 5 per md.	4,000	0	0	
Pigment	200 mds @ Rs. 40 per md.	8,000	0	0	
Cloth	150,000 yds. @ As. 6 per yd.	56,250	0	0	
Establishment	50,000	0	0	
Total				...	1,40,150	0	0
Rejects and other incidental losses 10%				...	14,015	0	0
Total cost for 150,000 yds.				...	1,54,165	0	0

∴ cost per yard 52"-54" wide Re. 1-0-6 approximately.

CHAPTER XVI

MISCELLANEOUS APPLICATIONS

1. VULCANISED SHELLAC

The question of the incorporation of accelerators, with a view to improving the properties of shellac, has been for some time past engaging the attention of most shellac research centres.

Vulcanisation of shellac formed the subject of an investigation at the Indian Lac Research Institute as a result of which it was found that the inclusion of 4-5 per cent of sulphur on the weight of lac and heating the mixture at 170°-180°C. for about half an hour showed an improvement of shellac in regard to its varnishing and moulding properties. The sulphurised or vulcanised shellac also proved superior to ordinary shellac in water-resistance, resistance to abrasion, scratch hardness and general mechanical strength. Products moulded from this modified shellac were also found to resist 'arc-ing' much better than ordinary shellac, presumably due to the presence of the non-conducting sulphur.

The sulphurised shellacs possessed the further advantage of giving bright-coloured varnishes and mouldings of any desired shade with the incorporation of comparatively small quantities of dyestuffs.

2. SHELLAC-RUBBER

A recent application of shellac has been the employment of small quantities of it to confer better properties on vulcanised rubber. It has been reported that rubber containing shellac is being employed by certain manufacturers for the making of the following articles with improved properties :

- i. Rubber soles and heels ;
- ii. Rubber flooring, mats and tiles ;
- iii. Moulded goods, toys, etc ;
- iv. Rubberised fabric ;
- v. Rubber-covered cables, tubing, etc.

It is believed also that a certain amount of shellac is used in the rubber tyre industry ; the increasing use of shellac in these directions would serve to give a new fillip to the demand for shellac.

The preparation of a rubber composition containing 10-20 parts of shellac for every 100 parts of crude rubber is the subject of a recent American patent. This addition of shellac to rubber and subsequent vulcanisation of both with sulphur are stated to increase the tensile strength, hardness and toughness of the vulcanised rubber and improve its ageing qualities. The shellac-rubber composition remains elastic after vulcanisation but harder and tougher than the corresponding composition containing only rubber. The inclusion of shellac is also claimed to reduce the porosity of rubber,

increase the resistance to oil and petrol, enhance the insulating properties and impart a shining surface which does not mar so quickly with ordinary handling as a pure rubber compound.

The main points which are to be taken into account in the manufacture of shellac-rubber are stated to be the employment of preferably only acid accelerators or a preponderance of acid accelerators if small quantities of basic accelerators are also used and the extra addition of about 3 per cent sulphur on the weight of lac for the vulcanisation of the latter. The accelerators suggested are 'captax' (mercaptobenzo-thiazole), butyl zimate (zinc dibutyl dithiocarbamate), 'altax', etc. The fillers and other ingredients are the same as for the vulcanisation of pure rubber.

The typical formula given in the patent is reproduced below for information :

Rubber	100 parts
Zinc oxide	5 "
Whiting	50 "
Carbon black	3 "
Captax	1 part
Stearic acid	1 "
Neozone D	1 "
Sulphur	3 parts
Shellac	10-20 "

3. SULPHITED LAC

Sulphitation of lac or the combination of sulphite radicles with the lac molecule has been investigated by the London Shellac Research Bureau. Lac can be dispersed in an aqueous solution of sulphur dioxide and the emulsion stabilised by the addition of a small proportion of stabilising agents like urea and triethanolamine. These dispersions of lac may be used for polishing furniture as a substitute for spirit varnishes. They can be applied by brush or pad and may also be used on plaster, metal and other surfaces. After the escape of sulphur dioxide, these emulsions leave a hard film insoluble in water and the elasticity of the film may be improved by plasticising it with urea, glycerine, etc. Baking at 120°C. has also been found to improve the adhesion, flexibility and hardness of the film considerably.

Sulphitation of lac may also be carried out either by treating a solution of lac in sodium carbonate solution with alkali bisulphites or by dissolving lac in an aqueous solution of sodium or ammonium bisulphite at a temperature of 60°-80°C.

The sulphited lac obtained by the hot bisulphite process has somewhat different properties from those obtained by either of the cold processes in that it is soluble in water but on baking, the water-soluble film becomes harder, tougher and glossier.

One of the important possibilities for the use of sulphited lac is in the manufacture of water paints and the product obtained by the hot bisulphite process is to be preferred for this purpose. The film hardens after continued air exposure and the

insolubility of the film is also found to develop by pigmentation and by incorporation of some drying oil in the mix. A typical formula for such a paint is :

- 100 parts of bisulphite lac (containing 30 per cent lac and 4-5 per cent ammonium sulphite)
- 30 parts of linseed oil.
- 380 parts of lithopone.

This composition has been suggested as a white road paint and is found to dry in 15-30 mins. on a frosty morning.

It has been claimed that sulphited lac made with sulphurous acid can find a promising use in plastic moulding. The use of a solution of sulphited lac will avoid the considerable power required in grinding and mixing of hot-mixed moulding powders and it is a much cheaper medium for wet mixing than alcohol. It would also reduce the time of after-cure and eliminate the use of accelerators for pre-curing on the rolls. Pigments not affected by sulphur dioxide may be incorporated during wet-mixing and those which react with the gas added after drying. The solution may also be treated with sodium hypochlorite when a white moulding powder is desired.

4. BOTTLE-WAX

By modifying shellac by treatment with a non-drying oil and lac acids under appropriate conditions tough and elastic products are obtained which could be used as sealing compositions. Mineral fillers may be added for lowering costs.

A typical composition which has been tested and found useful is made as follows :—

100 parts of shellac are mixed with 100 parts of hydrolysed lac and 10 parts of castor oil and the whole heated with stirring at 120°-130°C. for 2 hours over an oil bath. The melt is then poured and cooled when it sets into a fairly hard and plastic mass. The application of this sealing composition to corked bottles is made by melting the compound in a suitable vessel and dipping in the corked mouth of the bottles.

Another composition suitable for topping corks can be made by heating together 100 parts of lac, 100 parts of hydrolysed lac, 100 parts of pigment and 10 parts of castor oil.

5. SHELLAC ADHESIVE TAPES

A new application for shellac has been found in the preparation of adhesive tapes used in the electrical industries. As the result of various experiments, two compositions have been developed which are described below :—

100 parts of hydrolysed lac and 24 parts of linseed oil or castor oil fatty acids are heated at 150°-160°C. for 2 hours over an oil bath with efficient stirring, at the end of which 20 parts of glycerine are added and the heating continued at 140°-150°C. for another 3 hours to reduce the acid value from 140 to 80. The viscous mass is then cooled to 50°C. and weighed. The mass is then taken up with twice its weight of

denatured alcohol or rectified spirit. The resultant solution is then mixed with 25 parts of finely ground pitch and 1 part of an alcohol-soluble black dye and applied to cotton tape by dipping or brushing. The prepared tape is then allowed to be air dried before reeling.

Cotton tapes coated with the above composition have not lost their adhesive property even after keeping for a year.

6. INTERACTION OF SHELLAC AND SHELLAC ACIDS WITH ORGANIC ACIDS AND ALCOHOLS

It has been recognised that shellac is mostly composed of hydroxy acids in the form of condensed esters, lactides or lactones. From the constitution of shellac so far understood, it can be said to contain five hydroxyl groups and at least one carboxyl group. The predominance of a large number of hydroxyl groups, free and combined, led to the idea of modifying shellac by esterification with several organic acids and subsequent reduction of residual acidity by combining the esters with mono- or polyhydric alcohols. Such combinations might have specially water- and heat-resistant properties, an expectation fully confirmed by the results of actual experiment.

Shellac was condensed with several organic acids like maleic, phthalic, succinic, adipic, suberic, malic acid etc. Later, phosphoric and boric acids also were included in the list, and useful products were obtained. The condensations could be brought about directly or in the presence of the solvents and non-solvents of shellac. The alcohols investigated for reducing the final acidity of the condensation products include glycols, glycerine, butyl alcohol, etc. The modifications possess various degrees of hardness, elasticity and adhesion to different surfaces. A typical preparation with adipic and suberic acids with simultaneous condensation with alcohols has given promise of an extended use of shellac for special translucent flexible materials which could be made tough and hard by subsequent heat curing.

100 parts of shellac (preferably dewaxed) were heated with 6.2 parts of ethylene glycol and 14.6 parts of adipic acid in a glass or aluminium vessel, fitted with a mechanical stirrer, over an oil bath kept at 170°–180°C. for about an hour and the melt poured out in porous moulds made of plaster of Paris. The composition with the mould is then baked slowly for 24–28 hours at gradually increasing temperatures from 90°C. to 120°–130°C. The final product after complete curing is hard, tough and translucent which could be machined, sawed and turned.

The use of such modified resins for making combs, tooth-brush handles, buttons, etc. is suggested.

7. SHELLAC LACQUER FOR BLACK IRON SHEETS

The use of lacquers for the protection of black iron sheet surfaces against rust due to weathering and the action of other materials is well known. Shellac forms a very satisfactory protective film on most surfaces but its brittleness makes it unsuitable for use where any degree of flexibility is desired, unless it is suitably plasticised. A

satisfactory composition for such purposes may be obtained by reacting lac, preferably dewaxed (10 lbs.), with linseed oil fatty acids (3 lbs.) and glycerine (2 lbs.) under the conditions described on page 65 and dissolving the resinous product so obtained in spirit (1 gallon) and pouring the solution into 3 gallons of water containing 0.8 lb. of ammonia (d. 0.888). The iron sheets after coating with this varnish (by brushing or dipping) and air drying must be baked at 90°-95°C. for 2 hours. The sheets thus prepared may be punched, bent, twisted, stamped and/or otherwise processed to form containers without any cracking or peeling off of the protective film. In addition to general use, the containers may also be used for packing foodstuffs like jams, jellies etc., without any deterioration of the food stuff or damage to the film.

8. PROTECTIVE COATINGS FOR ELECTRICAL APPARATUS AND RESISTANCES

It has been found that by first esterifying shellac or shellac acid e.g. aleuritic acid, with a monohydric alcohol and then reacting the same with polybasic acid such as maleic or phthalic anhydride in presence of suitable proportion of fatty acids, saturated or unsaturated, or their mixtures, compositions are obtained which when dissolved in industrial alcohol or methylated spirit give a varnish with improved properties as regards resistance to penetration of moisture under all conditions of humidity. Coatings made with such compositions after baking for 1-3 hours at about 140°C. have been found to possess improved properties over that of ordinary shellac varnishes in that they remain flexible and are immune to changing humid conditions. The main use of this varnish is in protecting electrical apparatus in general and resistances in particular, especially high resistances such as are used in measuring instruments, radio components, etc. For example, resistances coated with varnishes made from shellac, maleic anhydride and linseed oil fatty acids exhibited no departure from Ohm's law and no significant increase in the conductivity during 18 hours' storage in an atmosphere of 90 per cent relative humidity.

The improved shellac varnish is prepared as follows: Lac, preferably dewaxed lac (100 pounds), is esterified with methyl alcohol (25 gals.) containing concentrated hydrochloric acid (4-6 pounds) as catalyst by refluxing for 3-4 hours in a steam jacketed kettle. After esterification is complete, the excess of alcohol is distilled off and the sticky balsam-like ester is washed with boiling water to free it from the catalyst and dried. The ester is then further reacted with maleic or phthalic anhydride (7 pounds) and linseed oil fatty acids (34 pounds) at 150°-160°C. for 4-5 hours in an open kettle. The resinous composition thus obtained is then thinned with industrial alcohol or rectified spirit to the desired consistency before use.

The manufacture of this varnish does not require any special equipment or machinery save those necessary for the preparation of an ordinary baking varnish and already existing in any paint and varnish factory.

The details of the above process are the subject matter of Indian Patent No. 29194.

CHAPTER XVII

LAC CULTIVATION

SEX DIFFERENTIATION AND ESTIMATION OF CROP

A study of the larval growth of the lac insect has enabled differentiation of the male from the female lac insect from an early stage. After a certain stage of growth has been reached, the male lac cell with its oblong shape can easily be distinguished from the more or less round female cell, with the naked eye. As the quantity of lac produced by the male lac insect is negligible and it completes its full course of life much earlier than the female, which for all practical purposes is solely responsible for all the output of lac, this knowledge of differentiating males from females when imparted to the cultivator will prove helpful to him in making a proper estimate of his crops.

FORECAST OF EMERGENCE AND FULL YIELD OF LAC CROPS

Investigations on the life-history of lac insects have shown that the mother lac insect continues producing lac almost to the last day of its life. On the average, each healthy female insect in its full life in *Rangini* crops produces about 0.029 grams of lac and in *Kusmi* about 0.069 grams. Therefore, to produce half a seer (1 lb.) of *Rangini* and *Kusmi* lac about 15,655 and 6,580 healthy lac females are respectively required.

It follows, therefore, that the cultivator, to get the maximum amount of lac from his crop, should allow the crop to remain on the trees nearly up to the swarming dates of lac larvæ. To meet this condition and also to avoid weakening or wasting the progeny by premature or late cutting of the brood lac, a simple method of forecasting the emergence of lac larvæ has been evolved. There is to be found a yellow spot on the lac covering each insect at its anal region. As the insect approaches its complete maturity, it shrinks and deposits wax and eggs in this region with the result that the resin is seen through and the yellow now appears orange in colour and covers a considerably larger area. The forecasting method is based on these changes which can be followed with the naked eye. With a knowledge of this, a cultivator, depending on the number of trees he has to reap and infect, can cut his crop from 24 hours to 8 days before the emergence of lac larvæ. In fairly large areas, the *Katki*, *Baisakhi*, *Jethwi* and *Aghani* crops should preferably be reaped five days before the emergence of lac larvæ.

TRANSPORT OF BROOD LAC AND CONTROLLING SWARMING OF LAC LARVÆ IN UNFAVOURABLE SEASONS

The delivery of eggs by the female is controlled by climatic conditions. Temperature plays a chief part both in the delivery of eggs and swarming of the larvæ.

The larvæ inside the lac incrustation are inactive below a temperature of 20°C. (68°F.) and egg-laying practically ceases below 17°C. (62.6°F.) in summer and 15°C. (59°F.) in winter.

All the female insects subjected to lowered temperature, 17°C. in summer and 15°C. in winter, or below, retain egg-laying vitality for 4-12 days depending on the delivery stage at the time of subjection to lowered temperature and the season. The yield of lac and the percentage of females in the progeny are not adversely affected as a result of subjecting the mother to lower temperatures.

It is, therefore, possible under cold storage to send mature brood lac even to long distances involving a journey of 9-10 days without any detriment to the output of lac by the insects used as brood lac. Devitalisation and weakening of the mother insects and progeny is caused by premature cutting, and late cutting causes emergence of larvæ during transit in the absence of cold storage.

Larvæ, hatching from the eggs and remaining in the lac test of the mother, die after 4-5 days. If the atmospheric temperature during the oviposition period is unfavourable to oviposition and emergence *i.e.*, below 18°C.-20°C. (64.4°-68°F.), emergence of larvæ from the brood will naturally be delayed. Such a situation may arise during January-February, the maturing time of the *Aghani* crops, as it actually did happen in the *Aghani* crop season of 1928-29. Swarming of the brood lac under these circumstances can be induced as follows:

(a) If the day temperature rises above 20°C. (68°F.), tying the brood to the hosts to be infected, when there is bright sunshine, will cause emergence to occur.

(b) Maintaining the brood in a warm room at a temperature of 24°-28°C. (75.2°-82°F.) will cause emergence to occur. If the brood is then taken and tied to the host plants, swarming will cease, unless the day temperature exceeds 20°C. (68°F.) and only those larvæ which have already emerged will crawl on to the host plant. Continuous emergence will not occur. But emergence can be restarted by returning the brood to the increased temperature once again. Under these conditions, brood lac should be kept in bamboo baskets and not handled singly or in bundles of one or two.

(c) Subjecting the brood to a temperature of 2°-13°C. (35.6°-55.4°F.) for a period of 1-4 days will cause continuous swarming to occur 2-3 days after return of the brood to an atmospheric temperature above 13°C.

The importance of the above should further be realised from the fact that the fertility of the lac insects in the *Aghani* crop (January-February) is the lowest, and leaving the brood lac to ordinary conditions in unfavourable seasons might mean practically a total failure of the *Jethwi* crop (June-July) which mainly serves as a brood crop for the next *Aghani* crop.

Preservation of Brood lac and obtaining better yields in Summer from Ber and Palas.

(1) **BER**

Plum (*Ber*) is the most common and the second most widely cultivated host of the lac insect, *Palas* being the first. The quality of lac produced by it falls, along with others, in the second class. It has an advantage over *Palas* in that it can be used as an alternative host with *Kusum*, the first class host of the lac insect.

Though *Ber* is such an important host and used extensively to cultivate both the *Katki* (June-July to Oct.-Nov.) and *Baisakhi* (Oct.-Nov. to June-July) crops, very little of the *Baisakhi* crop on it reaches maturity, with the result that most of the crop on it has to be cut *ari* (immature) from the middle of April to the middle of May. So much so, that most cultivators do not get sufficient brood from their trees for infecting for the *Katki* crop. In other words, a cultivator in *Baisakhi* crop gets only half the lac crop from trees as compared with *Katki* and very little as brood lac.

The main causes of mortality in the *Baisakhi Ber* crop are :

- (a) Hot winds in summer months ;
- (b) Leaflessness of the trees in hot weather ;
- (c) Shortage of food supply both to the plant and the lac insect thriving on its sap.

To overcome these difficulties, partial pruning of *Ber* trees carrying *Baisakhi* crop, in the last week of January is recommended. It provides the plant with new leaves in the hot weather.

Experimental results of four years' work at Namkum showed that in the months of April, May and June respectively, the percentage of trees with old leaves (which are unfit to manufacture fresh food and protect the tree against the sun and hot winds) in the control was 64, 16 and 11, and in the partially pruned trees, 26, 1 and 1; the percentage of leafless trees in the control was 14, 43 and 37, and in the partially pruned trees 4, 10 and 7 only ; the percentage of control trees which put forth new leaves in large numbers was only 7, 23 and 30, while in partially pruned trees, it was 36, 69, 74.

The ratio of the brood lac obtained from the control trees to that from the partially pruned trees in the most severe hot weathers of 1933-34 and 1934-35 was 1 : 4.52 and 1 : 4.11 respectively.

In partial pruning, (i) branches and shoots under $\frac{3}{4}$ inch diameter not bearing lac, (ii) every sparsely covered branch and (iii) those mainly covered with male lac insects are to be cut flush. And so also (iv) about a third of the shoots thinner than the little finger of a man should be removed from branches mainly covered with female insects.

Removal of mainly male-bearing branches further helps to reduce parasitisation of the female lac insects, as the male insects are more developed than the females at this time and are also more parasitised.

(2) PALAS

Palas is the most widely prevalent lac host in India. It has long leaf-stalks, 4" to 7" in length. The lac insect thrives equally well on the leaf stalks and shoots and the proportion of length of infectable shoots to leaf stalks of a tree is 1:2. However, the leaf fall in Palas begins in February and during the leaf fall the leaf stalks with lac insects on them either generally get wasted or when the prices of lac are high they are collected and cut into fine bits to adulterate scraped stick lac. In other words the lac produced on the leaf stalks in the *Baisakhi* season is negligible and the lac larvae that sit on them unwittingly meet a premature and undeserved death.

At least two-thirds of the *Baisakhi* crop from Palas is cut *Ari* (immature) in April-May. The chief reason for cutting the crop *Ari* is that due to leaf fall, Palas is practically naked in the first half of summer season and therefore in hot districts the majority of female insects die due to drought and the low biological activity of the host. The result is that (i) the cultivator gets only about half the quantity of lac that he could get if the insects survived in normal numbers till maturity as they do in the *Katki* crop when such adverse conditions are absent; (ii) there is shortage of brood lac to infect the succeeding *Katki* crop and therefore a great disproportionate distribution of trees to grow the *Katki* and *Baisakhi* crops.

To get over the above difficulties, partial artificial defoliation of Palas trees before infection in October is recommended. It results in following advantages: (1) saving in brood lac by at least one-fourth the quantity required otherwise for each tree; (2) provides the trees with new foliage in April-May when it is ordinarily naked or at best with leaf buds only and thereby enables more lac insects to survive than otherwise.

The experiments are being carried out in Kundri Forest which is one of the hottest places in Bihar; the results obtained during the last 4 *Baisakhi* crop seasons fully bear out the hypothesis that less brood lac would be required for infection and a bigger and better brood lac crop could be obtained by artificial defoliation of the trees in October before infection.

The leaf and its stalk are ripe by the end of June, and by the middle of September the leaf stalks can be easily separated from the shoots. The defoliation should be done a few days before infection in October-November. As far as possible all the leaves with their stalks except the top three from each shoot should be removed. Wherever easily accessible it is best done either by clasping the shoot with right hand from innerside at the base of the top leaf stalk and pulling it down; thus all the leaves that are to be removed will fall in one stroke, or by pressing off each leaf-stalk at the base from top side. Wherever it is not easily approachable a handy stick or the handle of village-axe may be used and the leaf stalks with leaves removed by giving a mild downward stroke at the base of the topmost leaf desired to be removed. In this way generally all the leaf-stalks on either side of each shoot can be removed in one stroke. The stroke should be downward and parallel to the shoot.

Partial artificial defoliation effects saving in brood lac, and enables more *Baisakhi* brood lac to survive.

Removing most of the crop *ari* at a time should be avoided, and instead, only those branches on which the lac insects are actually dead, i.e. have a pitted and dry appearance, should be removed at fortnightly intervals from 15th April onwards.

Partial pruning will reduce the scarcity of brood lac in *Baisakhi* crop to a great extent, giving larger yields, and lead to a more equable distribution of trees between the *Katki* and *Baisakhi* crops, and thereby make lac cultivation on *Ber* more profitable than at present.

Ants and Lac Crops

Investigations on insects associated with lac have shown that ants, which the cultivator generally considers as one of the chief enemies of lac, are not in general injurious to the lac insect, though some species undoubtedly pick up the crawling lac larvæ and the males at the time of their respective swarming, and this little amount of damage is of negligible importance. On the other hand, all the ants so far investigated and generally associated with the lac insect, are useful as they remove the excreted 'honey-dew' which otherwise mixed with dust, in the absence of rains, is likely to block up the breathing organs and anal opening of the lac insect, and thus cause death by suffocation. In addition, some of them, especially the big black ant named *Camponotus compressus* and the small red ant *Solenopsis geminata*, help the lac insect by feeding on the larvæ of the enemy moths and also picking up enemies from the scraped lac in godowns.

Insect Enemies of Lac

Researches have further revealed that there are two groups of enemy insects which attack lac crops. These are parasites and predators. The parasites are small winged insects belonging to the family *Chalcidoidea*; they lay their eggs inside the lac test either in or on the body of the lac insect; the young ones of these feed on the lac insect only and not on the lac produced by it. The damage done by this class varies from 5 to 10 per cent.

Predator damage is far more serious, the two insects mainly responsible being *Eublemma amabilis* (the white moth) and *Holcocera pulverea* (the blackish grey moth). The damage done by the larvæ of these insects amounts to about 35 per cent of the lac cells. The larvæ of these insects feed both on the lac insect and the lac produced by it.

The third predator which does a good deal of damage to lac insects especially in the second and third instars is *Chrysopa* sp. This predator walks over the lac encrustation and thrusts its sharp tube-like mouth parts into the body of the lac insect and sucks its body juices. The damage caused by this insect is not easily noticeable.

Control of Insect Enemies

Control methods can be broadly divided into 'Artificial' and 'Biological'. The following artificial measures are recommended :

1. Lac intended for use as brood should be cut as near to the swarming time as possible, never more than a week before, for optimum results.
2. In choosing lac for use as brood, healthy lac showing the minimum of parasite and predator attack should be selected.
3. Lac tied to the trees as brood should be removed as soon as the tree is sufficiently covered by the lac larvæ and in no case should it be allowed to remain on the trees for more than 3 weeks, because most of the lac larvæ swarm out by this time and thereafter the parasites and predators present in the brood would continue to emerge and attack the new crop.
4. Natural infection (*i.e.* leaving a certain amount of lac called *cheenti* on the tree to swarm *in situ*) should be abandoned particularly in October-November. Exception to this rule may be necessitated in certain localities, *e.g.*, infection of *Palas* in Palamau is said to be more successful with natural infection than with artificial.
5. All lac cut from the trees not required for brood and all brood lac after use should be scraped from the stick at once. This action alone destroys many of the larvæ and pupæ of the enemy insects and exposes others to climatic factors and to the attack of ants. Larvæ and pupæ of all insects which are easily visible may be crushed or killed by dropping in fire or in hot water.
6. If possible, the manufacturers, soon after purchasing the stick lac, should convert it into *chauri* (seedlac). This eliminates the parasites and predators which after emerging as adults try to escape into fields. Predators also breed on the stored lac.
7. In general, it is unwise to grow *Kusmi* lac in areas which are predominantly *Rangini*, or *Rangini* lac in areas which are predominantly *Kusmi*, as the *Kusmi* and *Rangini* crops overlap and become a source of natural infection of predators and parasites.
8. When a crop is cut all the lac sticks not required for brood lac should be tied into bundles of convenient sizes with a fairly heavy stone or brick and immersed under water in a river or stream or pond for 3-4 days and after that they should be removed and dried in shade and scraped. The Phunki brood lac and *Ari* lac should also be treated in the same way.

Biological Control is the control of insect enemies by their parasites. During the last four years control of *Eublemma amabilis* by *M. greeni* was tried on an experimental scale. The results indicate that by breeding *M. greeni* in the laboratory and liberating them in the crop from time to time the parasitisation of *Eublemma* by *greeni* can be increased to 100% more than is normally found. These require further investigations.

APPENDICES

APPENDIX I

STANDARD SPECIFICATIONS OF THE UNITED STATES SHELLAC IMPORTERS ASSOCIATION, INC., AND THE AMERICAN BLEACHED SHELLAC MANUFACTURERS ASSOCIATION, INC.

Seedlac

Seedlac is furnished in three grades, namely, *Koosmie* (also spelled *Kusmi* and *Koosmi*) *Baisakhi* (also spelled *Bisacki*, *Bysacki*, *Baisacki* and *Bysacky*) and Rangoon.

Seedlac shall conform to the following requirements :

		<i>Kusmi</i>	<i>Baisakhi</i>	Rangoon
Iodine number (on lac free from alcohol insoluble matter), maximum	...	18.0	18.0	18.0
Matter insoluble in hot 95 per cent alcohol, maximum per cent	...	3.0	5.0	5.0
Moisture, maximum per cent	...	2.0	2.0	2.0
Matter soluble in water, maximum per cent	...	1.0	1.0	1.0
Wax, maximum per cent	...	5.0	5.5	5.25
Ash, maximum per cent	...	1.0	1.0	1.0
Color	...	The color shall be equal to that of a sample mutually agreed upon by purchaser and seller.		

Dry bleached shellac

Dry bleached shellac shall be furnished in two grades, namely, regular and refined. Dry bleached shellac is white shellac. It is known as 'Bone-dry,' 'Vac-dry' or 'Kiln-dry' bleached shellac. The refined variety has had practically all the wax and matter insoluble in hot alcohol removed.

Dry bleached shellac shall conform to the following requirements :

		Regular	Refined
Iodine number (Wij's) maximum per cent	...	10.0	10.0
Matter insoluble in hot alcohol per cent	...	1.0	0.2
Moisture, maximum per cent	...	6.0	6.0
Wax, maximum per cent	...	5.5	0.2
Matter soluble in water, maximum per cent	...	1.0	0.3
Ash, maximum per cent	...	1.0	0.3
Color	...	The color shall be equal to that of a sample mutually agreed upon by purchaser and seller.	

APPENDIX II

MANUFACTURE OF MELAMINE

Calcium cyanamide (commercial) is extracted with approximately five times its weight of water at 45°–50°C. for 30 minutes. It is then filtered and the filter cake washed thoroughly with water. The total filtrate is separated into two equal portions, one portion cautiously neutralised with dilute sulphuric acid and then the two portions again mixed. This is then rapidly raised to about 75°C. and the acid required for complete neutralisation added at a constant rate for two hours. The calcium sulphate is then filtered off, washed and the clear solution concentrated to crystallisation. Dicyandiamide separates out as long needles (M. P. 210°C.).

Dicyandiamide (1 lb.) is heated together with strong ammonia (1.20 lbs.) in a high pressure steel bomb at 120°C. for 2½–3 hours. After cooling, the pressure is released, and the contents of the bomb repeatedly washed with cold water by decantation. The crystalline residue is recrystallised from boiling water when it separates as colourless shining plates, M. P. 350°C. Yield 0.3 lb.

The decanted aqueous solution is boiled for about two hours, allowed to cool and stand overnight. The precipitated impurities are filtered off and the clear solution evaporated to crystallisation. A tiny crystalline product separates out. Yield 0.25 to 0.3 lb. Very soluble in water and alkaline in reaction, this product (guanidine) is a very valuable by-product in the melamine manufacture as it has been tried for the preparation of injection moulding powders and promising results have been obtained.

APPENDIX III

PREPARATION OF LAC FROM KIRI

Kiri, as is well-known, is a by-product of the indigenous shellac manufacturing industry and the annual production of the same can be taken at 7 to 10 per cent of the total shellac manufactured. The average production of *Kiri* during the period 1920–1940 was about 40,000 maunds per annum and practically all this quantity used to be exported to Germany where the lac was recovered and marketed as Garnet Lac or under other trade names. It is well-known that the recovery process consists in dissolving powdered *Kiri* in rectified spirit, filtering away the impurities, recovering the solvent through distillation, and rolling the residual lac into thick sheets called Garnet Lac. Although the main principles of the process are well-understood, certain essential details in obtaining a standard high quality lac have been developed by the manufacturers and kept as closely guarded secrets. At the Institute, attempts were made to overcome a difficulty in the process, namely, the retention of alcohol in small traces in the final residue which makes the product sticky and the varnishes made therefrom slow-drying. Another difficulty was the rapid insolubilisation of the recovered lac during storage.

Under the present conditions in India when the cost of rectified spirit or methylated spirit has gone up very high and great difficulties are experienced in putting up solvent recovery plants, it was found necessary to fall back on the simpler process of recovering lac from *Kiri* by the use of alkali and acid. Under normal conditions, the alcohol process would no doubt be cheaper and give a more uniform product, but under present conditions in India it has been found cheaper and more practicable to adopt the alkali-acid process, which is described below in brief.

80 pounds of *Kiri* are powdered to pass through a 10-mesh sieve and stirred up in 40 gallons of water in which are dissolved 7 pounds of soda (sodium carbonate) in wooden vats and the temperature is raised to 70°C. by means of steam coils. When dissolution of the lac is complete, the solution is allowed to cool and filtered through muslin cloth. The filtrate is acidified with dilute sulphuric acid (diluted 1 : 5), added gradually till all the lac is precipitated. The precipitated lac is then washed several times with clean water until it is acid-free, the washing and filtration being carried out through muslin cloth. The acid-free lac is then centrifuged or pressed, to eliminate as much water as possible and then air-dried in thin layers. The product contains 5-6 per cent insoluble materials that are fine enough to pass through the muslin cloth used for filtering. A maund of *Kiri* normally requires 7 pounds of soda ash and 2.5 pounds of concentrated sulphuric acid and the yield is between 40 and 45 pounds.

Lac recovered as above from *Kiri* has been found suitable for the manufacture of compression moulding powders and many varnishes.

APPENDIX IV

PREPARATION OF HYDROLYSED LAC

25 pounds of shellac are dissolved in 12 gallons of water containing 5 pounds of caustic soda in a gas or electrically heated steel vessel. The solution is then boiled for 6-7 hours and allowed to cool. The shellac wax which floats to the top and forms a scum is removed by means of a ladle. The solution is then filtered into a wooden vat through muslin to remove off any impurities. The saponified lac or lac acids are precipitated from the filtrate in the form of sticky resin by the gradual addition with stirring of 2-3 gallons of dilute sulphuric acid (20 per cent). The precipitated lac acid is then washed with several changes of boiling water till the washed water is free from sulphuric acid. This is tested by the addition of barium chloride solution previously acidified with dil. hydrochloric acid to the washed water till the latter shows no turbidity or precipitation. The saponified lac is then dried free from moisture by heating at 100°-105°C. for about 3 hours. The yield of lac acids obtained is between 60 and 70 per cent.

APPENDIX V

TECHNICAL PREPARATION OF ALEURITIC ACID, $\text{CH}_2\text{OH} \cdot (\text{CH}_2)_5 \cdot \text{CHOH} \cdot \text{CHOH} \cdot (\text{CH}_2)_7 \cdot \text{COOH}$

Amongst the products of shellac hydrolysis with alkali, aleuritic acid is one of the components whose constitution has been definitely established. It has been found in this laboratory to be a valuable starting material for the preparation of transparent water-clear adhesives (as described above), plasticisers and resins in combination with other chemicals. Of the methods already reported in the literature and several others investigated in this Institute, the following has been found feasible for the preparation of aleuritic acid on a technical scale.

Shellac is hydrolysed by warming with 0.5 N potassium hydroxide. The quantity of potassium hydroxide required is determined from the saponification value of the shellac sample. To the solution of shellac in alkali, 1 lb. of sodium bicarbonate per 10 lbs. of shellac is added. This addition of sodium bicarbonate helps the calcium salt of aleuritic acid to remain in solution, while other acids are precipitated (as calcium salts) on subsequent addition of an acidified solution of calcium chloride. The calcium salts of liquid acids, as precipitated above, are filtered. The filtrate is acidified with dilute hydrochloric acid whereby aleuritic acid is precipitated. This is purified by dissolving in a mixture of alcohol and ethyl acetate (50:50) and crystallising.

The melting point of pure aleuritic acid is $100^\circ\text{--}101^\circ\text{C}$.

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